CONTRACT SERIAL NO. 89090-01-SS
REQUISITION NO. 23440-8-9090
CONTRACTOR: ST. MARY's UNIV.

PROJECT MANAGEMENT AND SCIENTIFIC SUPPORT FOR THE CANADIAN ADVANCED FLUIDS RESEARCH GROUP

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15 pp

PROJECT MANAGEMENT AND SCIENTIFIC SUPPORT

FOR THE

CANADIAN ADVANCED FLUIDS RESEARCH GROUP

A Report Submitted to

ENERGY, MINES AND RESOURCES CANADA

CANMET - ERL DIVISION

by

Dr. JOHN C. O'C. YOUNG

in fulfilment of

SSC Contract No. 23440-8-9090/01-SS
(SSC File No. 064SS.23440-8-9090)

December 21, 1989

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INTRODUCTORY NOTE

The following report of project management activities and scientific support services to the Canadian Advanced Fluids Research Group (CAFRG) discharges the contractual obligations of Dr. John C. O'C. Young and Saint Mary's University to Supply and Services Canada and Energy, Mines and Resources Canada (CANMET - ERL Division) under Contract No. 23440-8-9090/01-SS (SSC File No. 064SS.23440-8-9090) dated September 21, 1988 (the writer's birthday). Following several extensions and amendments, this contract was scheduled to be completed by December 29, 1989 (Raymond, 1989(b) - see appended list of references).

The "Statement of Work" contained in Appendix A of this contract (Straus, 1988) calls upon the contractor to:

i) act as the lead contact for the IEA Advanced Fluids Experts Group, participate with other international scientists from other IEA countries (particularly the USA and Denmark), and negotiate scientific aspects of the Advanced Fluids project on behalf of Canada;

ii) prepare and circulate progress reports on the IEA projects to the Canadian R & D group;

iii) act as project manager for the IEA project on ice slurry transport which has been awarded to Canada and ensure that work is done in a timely fashion and of appropriate quality for submission to the IEA Experts' Group;

iv) given the related developments by industry in North America and Europe, maintain liason with pertinent international chemical companies to assure that we have access to the latest developments in FRAs. Arrange for the negotiation of sample acquisition and testing as required;

v) provide scientific advice to CPDL to ensure that we do not infringe on existing patents and that we can negotiate access to all chemicals on behalf of Canadian users at reasonable commercial terms. Identify opportunities for development of new additives which are not covered by existing patents; particularly in the area of additives which may be suitable for use at temperatures below +10 deg C;

vi) conduct literature searches and such enquiries as are necessary to provide scientific support to the Canadian project team to resolve problems encountered during the R & D or applications engineering program. The results of such enquiries will be documented for circulation to the R & D team and for inclusion in a final report;

vii) in the Saint Mary's University test loops, conduct screening tests to select the most promising friction reducing additives (FRAs) for testing at the NRC or AECL test loops. This will include modification of loops so as to permit testing at low temperatures below +10 deg C;

viii) assist in the production and presentation of papers to report on work conducted by the project group, particularly the International District Heating and Cooling Association (IDHCA) annual meeting in July 1989;

ix) liase with other equipment and chemical suppliers whose participation might benefit our progress and provide such information as is required to familiarize them with the project objectives and opportunities;

x) produce summary reports to the IEA on the Canadian contribution which would comprise of submissions from the participants in the project group and may involve a progress report to the Executive Committee in November 1988 or May 1989; and

x i) compile all information, notes and related material into a report with an appropriate table of contents. Produce a summary report detailing findings and summarizing major developments. The report should include a review of progress by the Canadian Advanced Fluids R & D Group for the two year period leading up to this contract.

Tasks i) through x) have all been completed reasonably successfully and in a timely fashion, and this report completes item xi).

This report opens with a summary of project management activities under these ten tasks, a summary scientific progress report on the activities and achievements of the CAFRG through December 1989 and a set of recommendations for its future activities.

The body of the report consists of project management and scientific progress reports and a listing of supporting references (copies of which are available upon request).

SUMMARY REPORTS AND RECOMMENDATIONS

PROJECT MANAGEMENT SUMMARY REPORT

In April 1986, the United States Department of Energy (Kasza, 1986) submitted a proposal for the "Establishment of a District Heating and Cooling Advanced Transmission Fluids Experts Group" to the International Energy Agency (IEA).

During the summer of 1986, Wiggin of Energy, Mines and Resources Canada (CANMET) successfully solicited the interest of various parties in a Canadian involvement in the development of advanced energy transmission fluids for use in large-scale heating and cooling systems. The immediate aim of this initiative was to increase the energy carrying capacities (and/or to reduce the required diameters) of pipelines in water-based cooling systems by i) the use of chemical additives to reduce fluid friction losses, and ii) the use of pumpable ice slurries in place of chilled water (Wiggin, 1986(a) and 1986(b)).

In October 1986, Young was awarded a management contract "to act as the Canadian representative for the IEA project" and, amongst other responsibilities, "to brief interested parties on the project and determine the extent of their interest and capability to do work related to this research" (Guay, 1986).

Towards these ends, the following tasks and activities were carried out by the contractor:

> Under contract task i), he represented Canada at meetings of the IEA Advanced Fluids Experts Group in Canada, Denmark and the United States, and made numerous informal contacts with group members between these meetings. He participated in negotiations leading to two awards and one pending recommended award of IEA research contracts to members of the Canadian Advanced Fluids Group (CAFRG).
>
> Under task ii), he prepared and circulated (approximately) quarterly reports on IEA project-related activities to CAFRG members.
>
> Under task iii), as project manager of the IEA ice slurry project, he led and participated in progress review and planning sessions, prepared background technical assessments and literature reviews, wrote regular progress reports for the IEA and CAFRG members, and wrote the IEA Project Final Report on the " Hydraulic Characteristics of Ice Slurries and Chilled Water Flows" (which also forms part of this report).
>
> Under task iv), and in addition to regular contacts and discussions with industrial members of the CAFRG, he established and developed relationships with several international manufacturers of friction reducing additive (FRA) components; including, in particular, Akzo Chemicals Inc. of Chicago, Illinois, which donated almost all of the

surfactant compounds used in the test program and provided much valuable advice.

Under task vi), he prepared and circulated to CAFRG members computer-based literature searches on various project-related topics, including novel methods of ice-making, ice slurry hydraulics, FRA chemistry and FRA rheology.

Under task vii), he supervised the participating Saint Mary's University Research Group which, amongst other activities, developed and flow tested novel FRAs for low temperature applications and provided direction for their larger scale testing in the facilities of the Low Temperature Laboratory of the National Research Council of Canada.

Under topic viii), he prepared and presented reports or papers on research activities of CAFRG members at three International Energy Agency (IEA) Advanced Fluids Experts Group meetings, three scientific/technical conferences (including the 1989 annual conference of the International District Heating and Cooling Association (IDHCA), as stipulated in the contract) and various meetings of the CAFRG.

Under task ix), he established ongoing relationships with ice-making equipment manufacturers; corrosion inhibitor, surfactant and organic chemical manufacturers; and various research laboratories and other institutions with interests in the field.

Under task x), he produced four quarterly progress reports; two interim reports for meetings of the executive committee of the IEA Programme of Research, Development and Demonstration on District Heating; and a final report to the IEA on the Canadian 1988-89 IEA research project on "Hydraulic Characteristics of Ice Slurries and Chilled Water Flows" (which also forms part of this report).

Under task xi), he produced this report.

SCIENTIFIC PROGRESS SUMMARY REPORT

Following an initial period of goal setting, membership recruiting and background information gathering, the Canadian Advanced Fluids Research Group (CAFRG) was formed on July 27, 1987.

The CAFRG is an informal consortium of some twenty Canadian government agencies, national and provincial scientific laboratories, universities and private companies which was formed under the auspices of Energy, Mines and Resources Canada (CANMET - ERL) to develop and promote Canadian involvement in the development and application of district heating and cooling technology.

The CAFRG was formed, and has operated with considerable success, as an "R & D Club" in which the members work to i) produce new technologic developments of common interest and ii) promote the existing technologies and services that each member brings to the group. All members possess expertise relating to district heating and cooling, coupled with significant scientific and/or commercial interest to justify their participation (Wiggin, 1989(a)).

The CAFRG was represented by Wiggin and Young at the inaugural meeting of the International Energy Agency's Experts Group on Advanced Energy Transmission Fluids held on September 2-4, 1987. Despite a predominance of interest in district heating over cooling, the Canadian representatives were successful in promoting the cause of low temperature advanced fluid development and thus ensuring its inclusion in the program of activities recommended to, and accepted by, the International Energy Agency (IEA). Following this acceptance, a request for proposals was issued by the U. S. Department of Energy, acting as Agent for the IEA.

For its proposal of a study of "Hydraulic Characteristics of Ice Slurry and Chilled Water Flows", the CAFRG was awarded a contract on July 22, 1988 to carry out the low temperature component of the resulting IEA sponsored research program. Funds from this source, supplemented by EMR funding of management support services and contributions in "cash and kind" by CAFRG members, provided support for a first phase research program on low temperature advanced fluids which was completed in December 1989. While other CAFRG members afforded considerable assistance, the principal participants in this study were the Low Temperature Laboratory of the National Research Council of Canada, the Dearborn Chemical Company Limited and the Chemistry Department of Saint Mary's University.

When searches of the scientific literature indicated that no known friction reducing agent was likely to be effective at temperatures below 5 deg. C, the Saint Mary's University group created an additive testing laboratory and began its additive development program which eventually found that 0.1% w/w dosages of the surfactant/counterion pair n-dodecyltrimethyl ammonium 2,6-dihydroxybenzoate could produce pressure loss reductions in excess of 60% at temperatures down to at least -5 deg. C in 10% w/w aqueous ethylene glycol solutions over a pH range of 6 to 12.

Tests carried by the Dearborn Chemical Company Limited have shown that this additive is incompatible with certain types of commercial corrosion inhibitor, and studies of these undesirable interactions are continuing. It should however be noted that it is common practice in European district heating systems avoid the use of corrosion inhibitors altogether by purifying makeup water before use and then adding only mineral bases for pH control. It is likely that this practice will also gain acceptance in North America, and it is already being used for example in the Prince Edward Island Home District Heating System.

Using the proprietary ice slurry making unit developed by the Sunwell Engineering Company Limited (also a CAFRG member), NRC's Low Temperature Laboratory has shown that fine particle ice slurries are hydrodynamically indistinguishable from, or even preferable to, chilled water at the same temperature for ice solids concentrations up to 25% w/w. For an operating temperature range of 7 to 13 deg. C, the specific cool carrying capacity of chilled water is 25 kJ/kg , whereas that of a 25% w/w ice solids slurry is 84 kJ/kg neglecting any sensible heat contribution. This laboratory is currently investigating the benefits of combined use of friction reducing additives in ice slurries. Economic assessments of these benefits are also currently being made by the Energy Research Laboratory of CANMET, Energy, Mines and Resources Canada.

RECOMMENDATIONS FOR FUTURE WORK

The results of this study to date indicate that the combined use of friction reducing additives and pumpable ice slurries for district cooling applications (and the use of FRAs alone for district heating applications) constitute a very promising but immature and fragmented technology.

The potential capability of this technology to reduce substantially the capital and operating costs of the distribution component of district cooling (and heating) systems is implied in the foregoing "Scientific Progress Summary Report".

Ongoing efforts to achieve patent coverage of CAFRG FRA developments, to determine commercial-scale sources and manufacturing costs of additive components, and to assess the economic benefits of their use should be pursued expeditiously.

Environmental and toxicological issues relating to FRA use and disposal remain to be clarified.

The issue of corrosion inhibitor/FRA interaction remains unresolved, and the writer has already recommended EMR sponsorship of a joint Saint Mary's University/Nova Scotia Research Foundation study of this problem, together with an assessment of FRA corrosiveness in pure water systems not employing corrosion inhibitors (Young, 1989(j)).

The Saint Mary's University FRA development laboratory is well equipped to carry out flow testing studies on chilled and hot water media. Provision of a small ice slurry making unit for this laboratory should be considered.

The Saint Mary's University FRA development program should be re-activated to continue monitoring of developments in this fast evolving field, and to explore nonionic FRA development, the use of alternative counterion and surfactant components and dosage ratios, FRA use in methanol solution media, FRA durability under extended flow test conditions, FRA effects on heat exchanger and flow meter performance, alternative techniques for halogen ion removal from FRAs, development of an online FRA performance monitor etc., and to provide technical support services to the NRC ice slurry test program and the various planned FRA field trials.

Should it be called upon to do so, the Saint Mary's University research group currently does not have the resources to provide on-site technical support for these FRA field trials currently planned. Somewhat relatedly, the need of the writer's services as CAFRG program coordinator should be reassessed.

The NRC ice slurry test program should be extended to clarify the effects of ice solids characteristics and loading on hydraulic drag in pipelines, heat exchanger performance, and interactions with FRAs, and to provide supporting information for the proposed IEA/EMR project aimed at producing an ice slurry technology design manual.

Support of the CRIQ/Laval University effort to develop ice-making equipment to produce pumpable ice slurries from pure (glycol-free) water should be continued.

PROJECT MANAGEMENT REPORT

In April 1986, the United States Department of Energy submitted a proposal to the International Energy Agency (IEA) for the "Establishment of a District Heating and Cooling Advanced Transmission Fluids Experts Group" (Kasza, 1986).

During the summer of 1986, Wiggin of Energy, Mines and Resources Canada (CANMET) successfully solicited the interest of various parties in a Canadian initiative in the advanced fluids area; specifically relating to the development of chemical additives to reduce the fluid frictional resistance and/or enhance the volumetric energy carrying capacity of transmission fluids in district heating and cooling systems (Wiggin, 1986(a) and 1986(b)).

In October 1986, Young was awarded a management contract "to act as the Canadian representative for the IEA project" and, amongst other responsibilities, "to brief interested parties on the project and determine the extent of their interest and capability to do work related to this research" (Guay, 1986).

Following preliminary studies of the relevant technical literature, Young visited and discussed the advanced fluids project with some thirty potential participants in January, 1987, and followed up these contacts with a formal solicitation of their participation in February, 1987 (Young, 1987(a)). Addressees were provided with copies of a background literature review and were invited to select from a list of five potential R & D activities. Additional literature search materials were circulated on March 18, 1987 (Young, 1987(b)).

Based on the responses received, the initial membership of the Canadian Advanced Heat Transmission Fluids Research Group was established (Barringer, Boulanger, Butterworth, Jean, Larkin, Linton, Morofsky, Paradis, Snelson, Sukhwal, Waller, Wiggin, Young and Zaworotko). This group selected as its immediate priorities i) studies of gas hydrate formation by direct-contact heat exchange (7 votes), ii) development of additives to inhibit ice crystal adhesion and improve the bulk handling properties of ice slurries (6 votes), and iii) development of additives to reduce fluid friction and/or enhance heat transfer from heat/cool transporting fluids (5 votes). Young (1987(c)) reported these results back to the membership, provided them with the first of a series of literature review reports on these topics and proposed an initial meeting of the group for July 1987.

Young (1987(d)) reported the outcome of this inaugural meeting, which was held in Ottawa on July 27-28, 1987. The participants were Barringer, Boulanger (written submission), La, Larkin, Linton, Paradis, Snelson, Snoek, Waller, Wiggin and Young, and the following is a slightly condensed version of the report of this meeting:

i) Introduction and Opening Statements by Participants

Michael Wiggin, Energy, Mines and Resources Canada, (Project Scientific Authority)

Wiggin began by outlining the history of the project and referred to its underlying (Delphi-like) strategy of appointing a project coordinator (Young) to create an expert research and development team, and to reach a consensus on research priorities and prepare plans to achieve them by a "cooperative" process of stepwise information dissemination, consultation and solicitation of participant responses.

He suggested that we had been casting around rather broadly thusfar and that this meeting should attempt to reach a consensus on the project priorities of our group, to agree on required R & D activities and how they might be carried out. He further suggested that the most pressing need was for participants to provide guidance to the Canadian representatives (Wiggin and Young) who would be attending the upcoming meeting of the IEA's Experts Group on Advanced Transmission Fluids Research to negotiate Canada's involvement in this work.

John C. O'C. Young, Professor and Consulting Chemical Engineer, Saint Mary's University, (Project Coordinator)

Young stated that his immediate responsibilities to the project were to gather and analyze background materials relating to the five research topic areas which participants had thusfar identified as possible candidates for the group's involvement in the IEA's Advanced Transmission Fluids Project (Young, 1987(b)).

He summarized project activities to date and presented an update on his literature-searching activities since his Round Two Report (Young, 1987(c)).

In the ensuing discussion, Barringer referred to the possible use of insoluble additives such as air bubble injections and of mechanical boundary layer devices to break up turbulent eddies and so reduce friction losses. La referred to changes in the behavior of ice slurries upon aging, stated that pumping of slurries at 30-50% solids presents few problems but that more work needs to be done on handling slurries at 80% solids. He mentioned the use of ice slurry air conditioning for mine cooling and stated that vacuum freezing processes were currently being used in South Africa.

Chris Barringer, Energy Systems Centre, Ontario Research Foundation, (which is active in the development, analysis and testing of a broad range of space conditioning equipment)

Barringer stated that a number of groups at the Ontario Research Foundation had shown interest in the project. The Energy Systems Group is involved in fluid flow and heat transfer studies and has considerable experience of heat pump testing, modelling and monitoring. The Polymer Chemistry Group is currently engaged in developing friction-reducing coatings for application to underwater vessels, and has experience of how to manufacture and cross-link polyethylene.

Michel Boulanger, Head, Energy Group, Centre de Recherche Industrielle du Quebec (CRIQ)

Boulanger could not attend the meeting, but wrote to indicate his continuing interest in the project in general, and in absorption refrigeration, ice adhesion and freon substitution studies in particular (MB to JCO'CY, letter of July 7, 1987). In regard to the last of these topics, he stated that "Your suggestion (in Young, 1987(c)) on freon replacement projects points to an extremely important and timely subject. So important that it should be in itself a major international undertaking, separate from the limited scope of our present project. Here is an excellent opportunity for Canada to promote, and in which we (CRIQ) are ready to participate".

David La, Chief Engineer, Sunwell Engineering Company Limited, (manufacturers of thermal storage and ice-slurry making equipment)

La described his company's line of bulk freezing, ice-making equipment, and in particular its commercial (5-ton per tube) crystallizing heat exchanger in which a plastic screw wipes the inner tube surface to remove the chilled layer before freezing begins. He described a number of such systems which Sunwell has already installed,indicated that the company is also exploring other crystallizer designs, and stated that it was interested in participating actively in our development and evaluation of ice-making equipment for district cooling.

Brian Larkin, Senior Research Officer, Low Temperature Laboratory, National Research Council

Larkin stated his interest to be heat transfer in general and heat pipes and heat exchangers in particular.

Jeff Linton, Senior Research Officer, Low Temperature Laboratory, National Research Council

Linton indicated that his principal current research activity concerned a steam-generating heat pump test rig, but that he was generally interested in heat pump aspects of our project.

Marc Paradis, Professor, University of Laval, (research on ice-making heat pump technology)

Paradis outlined his work on the development of an ice-making heat pump in which water is supercooled on the tube side of the evaporator before disharge to the crystallizer.

Keith Snelson, Senior Research Officer, Low Temperature Laboratory, National Research Council

In addition to the steam-generating heat pump work referred to by Linton, a non-azeotropic refrigerant test rig will be starting up shortly in the Low Temperature Laboratory and Snelson also indicated an interest in the "Puraq" ethylene glycol-water absorption refrigeration process.

Chris Snoek , Head, Experimental Analysis Section, Thermalhydraulics Development Branch, Atomic Energy Company of Canada Ltd., (which has a large professional staff and extensive experimental facilities for making large-scale system evaluations of heat and mass transfer processes)

The Thermalhydraulic Group is engaged in two-phase flow studies and heat transfer and pressure drop measurement, and has "a fair bit of experience of running big rigs and water and freon loops", and is seeking diversification opportunities.

John Waller, Research Director, Dearborn Chemical Company Limited, (development and marketing of water treatment chemicals)

Waller stated that his company had a history of supplying corrosion inhibitors, scale inhibitors and microbiocides to district heating and cooling systems, and that it could best contribute to the project in the area of experimental evaluation of erosion and corrosion inhibiting additives for use with ice slurries.

ii) **Discussion of Project Targets and Participant Contributions**

The following technologies were selected as being of prime interest to participants:

- friction-reducing additives for both aqueous ice slurries and water for temperatures ranging from -5 to 120 °C
- the generation and transport of ice slurries in both closed and open systems (including, in the latter category, low temperature water source heat pumps which might recover the latent heat of fusion of ice by generating ice from surface water and delivering the recovered energy to space heating or similar systems)

The following applications/markets were perceived:

- **friction reducing additives**

  district heating and cooling
  building heating and cooling

- **ice slurry production/transmission**

  district heating and cooling
  building heating and cooling
  cold storage for air conditioning
  industrial and mine cooling
  ice for food and beverage industries
  concentration of toxic wastes
  water salination/purification
  cool surface water heat extraction

The following R & D requirements were suggested for friction reducing additive evaluation:

- **background studies to find potentially useful additives**

  friction reducing potential
  stability/degradability
  impact on heat transfer performance
  impact on heat and flow metering
  impact on corrosion and biological fouling
  interaction with other additives in heating/cooling systems
  toxicity of additives and/or their degradation products
  constraints on disposal after use
  impacts on performance of heat pumps, boilers,exchangers, etc.
  additive economics

- **evaluation of candidate additives**

  development of screening protocol and procedure
  pilot scale screening tests
  full-scale testing

The following R & D needs were suggested for the development of ice slurry generation & transmission systems

- **additive-related**

  interaction of friction reducing additives with ice-making additives
  impact of these additives on ice characteristics (structure,size etc)
  impact of these additives on supercooling tendencies
  effects of concentration changes during freezing and melting

- **Ice slurry production and handling**

  description of slurry production technologies

  - bulk freezing (Sunwell technology)
  - supercooling (Paradis technology)
  - direct contact heat exchange
    primary (vacuum flash) refrigeration
    secondary (freon/alkane) refrigeration

  characterization of slurry production technologies

  - pros and cons
  - stage and scale of development

slurry production development tactics

- technology improvement or development or acquisition or joint venture options

slurry handling and transmission

- friction vs. ice fraction
- influences of ice age, size distribution etc.
- pumping techniques and requirements
- influences of slurry generating additives on the above items
- interaction of ice additives with friction reducing additives
- impact of ice slurry particles on corrosion and erosion
- impact of ice slurry particles on corrosion protection films
- ice separation, stratification and entrapment in systems
- storage behavior and metering methods

iii) **Next Steps**

The meeting ended in agreement that the above items represented the priorities of our group, that Wiggin and Young should prepare position statements based upon them for presentation to the September meeting of the IEA's Expert's Group on Advanced Transmission Research, and that they should subsequently report back to our group as to what program of work would be most desirable for Canada, what we might do for the IEA Group, what might be a useful complementary R & D program and what commercial opportunities might exist in the field.

Postscript

In trying to match our capabilities with the aspirations listed above, it seems to me (Young) that the specific strengths of our group, in no particular order, are as follows:

- the commercial ice-making capability, associated testing facilities and related technical skills of Sunwell Engineering Co.

- the large-scale fluid flow and heat transfer monitoring pipe loops and other systems and related technical skills of AECL, plus potential access to several university heating and air conditioning systems for full-scale testing of additives and/or ice-making equipment

- the extensive experience and facilities for testing water conditioning additives of Dearborn Chemical Company

- the interest of ORF in friction-reducing coating development

- the fluid flow and heat pump performance evaluative experience of CRIQ, ORF and Laval University

- the refrigeration and refrigerant knowledge and test facilities of CRIQ and NRC's Low Temperature Laboratory

- the information sleuthing skills and interests in novel energy storage media of the Saint Mary's University group

Given these capabilities and resources, I suggest that we plan to develop projects in one or both of the following areas:

- the use of **friction reducing additives** in conjunction with **ice slurries** and their testing in **large-scale systems**

- participation in the broader problem of developing and evaluating environmentally benign **freon-substitute refrigerants** and pilot plant testing of such refrigerants in the context of **direct contact ice-making**

Following this founding meeting of the Canadian Advanced Heat Transmission Fluids Research Group, its representatives (Wiggin and Young) attended, as members, the Inaugural Meeting of the IEA Experts' Group on Advanced Energy Transmission Fluids which was held at Ohio State University on September 2-4, 1987. Reports of this meeting were prepared by both Young and Wiggin (Young, 1987(e)).

Participants agreed on priorities of i) information exchange (highest); ii) coordination of research activities; iii) applied research (laboratory and demonstration) activities; and iv) basic research work (lowest). After considerable discussion, the following topic areas were selected and prioritized (1 highest; 5 lowest) by the various national representatives as follows:

Topic	Type	Can	Den	Ger	Hol	Swe	USA	Total	Rank
1. Drag Reducing	Hot	2	1	1	1	1	2	8	1
2. Additives	Cold	3	4	4	4	4	1	20	3
3. PCM Transport	Hot	4	2	2	2	2	4	16	2
4. Systems	Cold	1	5	5	5	5	3	24	5
5. Novel Concepts	- - -	5	3	3	3	3	5	22	4

These ballot results showed a considerable initial divergence of opinion between the European participants on the one hand and Canada and the United States on the other. However, following further discussion of the current status of the technologies of these five topic areas, hot phase change materials (Topic 3; specifically high density polyethylene) was downgraded in priority, low temperature applications were elevated somewhat, and the following list of activity topics was prepared for prioritization:

1. preparation of a status report on friction reducing additives
2. coordination of research on friction reducing additives (FRAs)
3. corrosion inhibitor and other additive studies with FRAs
4. engineering data for components in FRA systems
5. friction reducing additive economics
6. coordination and reporting of FRA demonstrations
7. ice slurry systems
8. phase change materials for heating applications
9. novel concepts (clathrates, conjugating binary solutions, etc)
10. basic research on additives

For purposes of assigning priorities, FRA-related items 1, 2, 4, 5 and 6 were grouped together but item 3, which is also related to FRAs, was retained as a separate topic because of its particular importance. The following priorities and suggested IEA contribution levels were established:

Topic	Can	Den	Ger	Hol	Swe	USA	Total	Rank	$K
FRAs (1,2,4,5,6)	1	1	1	1	1	1	6	1	45
3. Corrosion	2	3	2	2	2	2	13	2	75
7. Cold PCMs	3	6	3	6	3	3	24	3	50
8. Hot PCMs	6	2	4	5	5	4	26	4	30
10. Basic FRA R/D	4	4	6	4	4	6	28	5	75
9. Novel Concepts	5	5	5	3	6	5	29	6	25

The project manager (Kaminsky, USA) undertook to prepare requests for proposals on i) the screening and testing of FRAs, ii) effects of FRAs on the performance of system components (fittings, valves, heat exchangers, etc.), iii) analysis of the economics of FRAs, iv) a feasibility study of transportable phase change materials (high density polyethylene), v) a novel fluids assessment, vi) a compilation of information on large-scale use of FRAs, vii) ice slurry transport and viii) basic R &D on FRAs. Young was asked to prepare a proposal on "novel concepts" (hydrates, conjugating binaries, clathrates, etc), namely item v), but declined after some consideration (thereby showing good judgment since the project was subsequently eliminated because of budget limitations).

As directed, Kaminsky (1987(a)) prepared a research plan based on the six project areas and proposed budgetary allocation tabulated above, for submission to the November 1987 meeting of the IEA District Heating and Cooling Executive Committee. Outlines of these project proposals were as follows:

PROJECT 1 DISTRICT HEATING FRICTION REDUCING ADDITIVES, RESEARCH COORDINATION AND INFORMATION EXCHANGE

The project will consist of six tasks

a. Status report of ongoing activities in advanced fluids

b. Compilation of additive screening and testing activities - temperature range; concentration; velocity; line size and length; water quality; pipe type, material, roughness, coating, etc.; duration; additive description - source, supplier, toxicity, friction reduction; heat transfer; longevity; corrosion; cost information

c. Engineering data for components

d. Economic analysis of additives

e. On-site demonstration summary report

f. A guide for the use of additives

PROJECT 2. FRICTION REDUCING ADDITIVES ___Side-effects

Corrosion and contamination by friction reducing additives and their interactions with other water treatment additives

PROJECT 3. DISTRICT COOLING _____ ICE SLURRY

The project will consist of four tasks

a. Influence of crystal size, shape and loading on pressure drop, heat transfer and handling characteristics

b. Assessment of pertinent literature

c. Effect of additives

d. Evaluation of system design options

PROJECT 4. PHASE CHANGE MATERIALS FOR DISTRICT HEATING

The project will consist of two tasks

a. Economic and technical analysis to assess feasibility

b. Identification of PCMs in the 90-129 deg C range

PROJECT 5. BASIC RESEARCH _____ On FRAs

This research may include

a. Establishment of the relationship between chemical structure, colloidal structure and friction reduction

b. Impacts of structure on friction reduction and heat transfer

PROJECT 6. NOVEL CONCEPTS

The project will consist of three tasks

a. Compilation of information on ongoing research to include
- hydrates - conjugate binary fluids -clathrates

b. Preliminary feasibility assessment

c. Identification of research requirements

As part of his followup activities to this request for proposals, Young solicited a proposal from Atomic Energy of Canada Limited (Snoek, 1987) for tests on drag reducing agents in a relatively large (10 cm diameter) instrumented pipeline system.

Young's project management contract was extended on November 26, 1987 (Davidson, 1987).

Following a notification that the the IEA Executive Committee was prepared to fund only $185K (US) of the budget of $300K (US), a decision was made to restrict the request for proposals to PROJECTS 2, 3 and 4 in the above list (Kaminsky, 1987(b)).

The second meeting of the Canadian Advance Fluids Research Group was held in Ottawa on January 14, 1988 to consider the group's next steps in regard to ice slurry making, ice slurry handling and friction reducing agents (Young, 1988(a)). The group decided to respond to the IEA project RFPs relating to hot water friction reducing agents and ice slurries (PROJECTS 2 and 3), and agreed to assist Young with the preparation of these submissions.

On January 19, 1988, Young (1988(b)) issued a review of the patent literature relating to FRAs.

In February 1988, Young (1988(c)) prepared and circulated to CAFRG members a literature review of ice-making technologies for space cooling

On behalf of, and in consultation with, members of the CAFRG, Young prepared research contract proposals on i) "Batch and Flow Testing of Interactions Between Friction Reducing Additives and Water Treatment Chemicals" and ii) "Influences of Ice Slurry Characteristics on Hydraulic Behavior", and submitted them to the IEA project manager on March 7, 1988 (Young, 1988(d) and (e)).

At Young's request, an assessment was made by CPDL of a series of patents relating to friction reducing additives which his literature searches had uncovered (Thomson, 1988).

In March 1988, Young attended a conference on the U. S. Department of Energy's Thermal Energy Storage Program in Starkville, Mississippi, at which papers were presented on CBI's direct-contact, oil-less compressor, ice-making process, Argonne's high density polyethylene work (withdrawn) and Stiel's work on conjugating binary liquids (Young, 1988(f)).

On April 10, 1988, Young (1988(g)) issued an assessment of the CPDL patent review (Thomson, 1988).

In April, 1988, Kaminsky (1988) recommended IEA support of the research proposal on ice slurries (Young, 1888(e)), but rejection of the proposal of work on interactions between friction reducing agents and water treatment chemicals (Young, 1988(d)).

In May 1988, Young initiated what proved to be a valuable contact with Sydney Shapiro, the Director of Technical Services of Akzo Chemie America, Chicago, Ill., who subsequently provided many chemical samples and much useful advice on surfactants (Young, 1988(h)).

The third meeting of the CAFRG was held in Ottawa on June 9-10, 1988 (Young, 1988(i)). A subgroup on ice slurry making agreed to support Paradis' supercooling ice-making technology, and recommended that the CAFRG should also promote demonstrations of both glycol-based (Sunwell) and non-glycol-based (tentatively Sabroe, see Boulanger, 1988) ice-making technologies. It also recommended that a study be made of the use of ice-making for freeze concentration in general and toxic waste concentration in particular. A subgroup on ice slurry handling received a program outline from Young on the development of FRAs for ice slurry applications. It was agreed that FRA/ corrosion inhibitor compatibility tests would be carried out by the Dearborn Chemical Company and ice slurry hydraulic testing would be performed in the Low Temperature Laboratory of the National Research Council in Ottawa..

A progress report on the Saint Mary's University group's low temperature FRA development work was presented to CAFRG members in July, 1988 (Young, 1988(j)).

Following some negotiation and acting on behalf of the CAFRG, Young (1988(k)) signed a research contract to carry out the IEA ice slurry and low-temperature friction reducing agent project.

On September 21, 1988, Young's project management contract with DSS/EMR was renewed, and his duties and responsibilities were redefined (Straus, 1988).

In September, 1988, Young specified and obtained industrial surfactant samples from Akzo Chemie America for experimental testing as friction reducing agent components (Shapiro, 1988) and arranged for FRA/corrosion inhibitor compatibility testing by the Dearborn Chemical Company (Young, 1988(l)). An initial progress report on this work was reported by Thompson (1988).

On November 8, 1988, Young (1988(m)) issued a first quarterly progress report on the IEA research project.

The fourth meeting of the CAFRG was held in Ottawa on December 8, 1988, and its activities were summarized by Young (1988(n)) and Wiggin (1989(a)).

On December 9, 1988, Young (1988(o)) issued an assessment of the patent situation relating to friction reducing agents.

On January 12, 1989, Thompson (1989(a)) issued a second progress report on his FRA/corrosion inhibitor compatibility work.

On January 20, 1989, Larkin (1989(a)) issued an initial progress report on his ice slurry work.

On February 25, 1989, Young (1989(a)) submitted his second quarterly progress report on the IEA project.

On February 28, 1989, Young received a grant under the EMR Research Agreements Program in support of his group's work on low temperature FRA development (Babcock, 1989).

On March 2,1989, Wiggin (1989(b)) provided supporting evidence for patent coverage of the Saint Mary's University's work on low temperature FRAs.

On March 5,1989, Young (1989(b)) reviewed progress of the ice slurry test program in NRC's Low Temperature Laboratory

On March 7, 1989, Young (1989(c)) reviewed progress of the FRA/corrosion inhibitor interaction test program.

On March 20, 1989, Young (1989(d)) specified the FRA for pilot-scale testing in NRC's Low Temperature Laboratory.

On April 5, 1989, Young (1989(e)) presented a paper on the CAFRG's work on FRA/ice slurry combinations to a meeting of the IEA's Advanced District Heating and Cooling Transmission Fluids Experts Research Group in Herning, Denmark on April 4-5, 1989.

On April 10, 1989, Young (1989(f)) requested and subsequently received supplementary funding from the IEA to purchase additional equipment for FRA testing in his laboratory.

On April 12, 1989, Thompson (1989(b)) issued a third progress report on his FRA/corrosion inhibitor compatibility work.

On May 8, Young received a report he had requested on water quality and FRA performance in the Herning (Denmark) district heating system (Hammer, 1989) and circulated it to CAFRG members.

On May 17, 1989, Young (1989(g)) issued a third quarterly progress report on the IEA research project.

In June 1989, Larkin (1989(b)) and Young presented a paper on their work on ice slurry behavior and interactions with FRAs at the 80 th Annual Conference of the International District Heating and Cooling Association in Virginia Beach, Va.

On July 5, 1989, Young (1989(h)) initiated an ongoing correspondence with M. W. Hardy and Co. Inc. of New York City concerning the possibility of that company acting as a commercial manufacturer of the surfactant counterion identified by the Saint Mary's University Group as being most effective for friction reducing applications.

On July 21, 1989, Young (1989(i)) wrote to Harvey Thompson of the Dearborn Chemical Company suggesting a "European approach" to the problem of solving the FRS corrosion inhibitor incompatibility problem.

On July 25, 1989, Larkin (1989(c)) issued a second progress report on NRC's ice slurry test program.

On July 27, 1989, Young (1989(j)) solicited a research proposal from the Nova Scotia Research Corporation relating to the FRA corrosion inhibitor incompatibility problem.

On July 25, 1989, Larkin (1989) provided a progress report on the ice-slurry study taking place in the Low Temperature Laboratory of the National Research Council in Ottawa.

Murray's Honors Thesis on "Micelle Forming Characteristics and Drag Reducing Capabilities of Selected Nitrogen Containing Surfactants at Sub-Ambient Temperatures" was published in August 1, 1989 (Murray, 1989).

On August 1, 1989, Young (1989(k)) wrote to Elizabeth Blackburn of CANMET outlining his thoughts on patent lawyer Norris Eade's comments on Murray and Young's patent application on low temperature friction reducing additives.

On August 3, 1989, Young (1989(l)) sent a memorandum to Harvey Thompson suggesting some new initiatives in regard to the FRA/ corrosion inhibitor interference impasse.

In August, 1989, the Saint Mary's University Group issued their report on "The Evaluation of Certain Surfactant/Counterion Combinations as Friction Reducing Additives" (Cleary, et al.,1989).

Wiggin and Young attended a meeting of the IEA Advanced Transmission Fluids Experts Group in Ottawa on September 14-15, 1989, for which minutes were subsequently provided by Kunjeer (1989(a)). Two research proposals for future IEA support were submitted on behalf of the CAFRG, namely i) to produce an ice slurry technology design manual and ii) to develop an online instrument for monitoring the condition of FRAs in pipelines. The Experts Group recommended support of the first, but not of the second, of these proposals. The other supported projects were "Environmental Effects of Surfactants" (University of Dortmund, FRG); "Plate Heat Exchanger Performance" (B & S Group, Denmark); "Influences of FRA Chemical Structure on Corrosion Rates" (Ohio State University, USA); and "Effects of pH on Pitting of Metals in Contact With FRAs" (Ohio State University, USA).

On September 30, 1989, Young (1989(m)) issued a fourth quarterly progress report on the IEA research project, and circulated a project summary report prepared for the November meeting of the IEA District Heating and Cooling Executive Committee.

In October, 1989, Young (1989(n)) prepared a report for CPDL presenting additional information in support of the patent application of Murray and Young relating to low temperature friction reducing additives.

On October 27, 1989, Young (1989(o)) presented a paper on his group's work on friction reducing agent development to the Nova Scotia Association of Science Teachers Annual Conference.

On October 27, 1989, Gupta (1989) provided a third progress report on the NRC Low Temperature Laboratory's ice-slurry study.

On behalf of the various project patricipants, Young (1989(p)) prepared and submitted a final report in November, 1989, on CAFRG's IEA project for inclusion in the overall project report to IEA (Kunjeer, 1989(b)).

In November, 1989, Young cooperated with Rob Brandon (1989) of the PEI Energy Corporation and Goran Mornhed of AFE Engineering in the preparation of a research proposal to Energy, Mines and Resources Canada relating to the testing of friction reducing agents in a district heating system.

On November 9, 1989, Young (1989(q)) submitted a proposal for funding under EMR's Research Agreements Program to study the effects of friction reducing additives on heat exchanger performance and corrosion inhibitor effectiveness.

On November 20, 1989, Young (1989(r)) submitted a proposal for joint NSERC/EMR funding under the latter's Research Agreements Program to study the effects of friction reducing additives on heat exchanger performance and corrosion inhibitor effectiveness.

On November 29, 1989, Young (1989(s)) submitted a research proposal to the Institute for Chemical Science and Technology, in which he asked for funding for a joint program of studies of interactions between corrosion inhibitors and FRAs to be carried out at the Nova Scotia Research Foundation Corporation and Saint Mary's University.

In December 1989, Young sat down to write this report . . . . .

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SCIENTIFIC PROGRESS REPORT

Reports on the progress of this project have already been presented in a number of forms, most if not all of which are listed in the reference section of the foregoing "Project Management Report". These include the various informal submissions made by various participants to CAFRG meetings, the memoranda and reports prepared by and circulated amongst CAFRG members, the conference papers presented by Larkin and Young, the thesis on FRA research prepared by Murray, and, most recently, the final IEA contract report submitted by Young on behalf of participating CAFRG members and those who assisted them with their efforts.

The following copy of this final IEA contract report also constitutes the writer's final scientific progress report under EMR/SSC Contract No. 23440-8-9090/01-SS.

HYDRAULIC CHARACTERISTICS OF ICE SLURRIES AND CHILLED WATER FLOWS

A Report prepared by Dr. John C.O'C. Young

of the Department of Chemistry

of Saint Mary's University

on behalf of the

Canadian Advanced Heat Transmission Fluids Group

November 3, 1989

HYDRAULIC CHARACTERISTICS OF ICE SLURRIES AND CHILLED WATER FLOWS

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SUMMARY

Small-scale experimental studies made by our various project participants indicate that the use of transportable ice slurries and hydraulic drag reducing agents can substantially reduce the capital and/or operating costs of district cooling systems and other refrigerative applications.

Ice solids loadings of up to 25% w/w were achieved with insignificant effects on the hydraulic and heat exchange properties of a glycol-water medium, and these ice slurries provided a multiple of the "cool" carrying capacity of an equal volume of chilled water.

A hydraulic drag reducing reagent (n-dodecyl trimethyl ammonium 2,6-dihydroxy benzoate) was developed specifically to meet the requirements of operation at temperatures down to and below the normal freezing point of water, and in excess of 50% drag reduction was achieved.

This drag reducing agent was shown to be equally effective over pH values ranging from 6 to 12, and should be suitable for use in cooling systems which employ deionized water, mineral bases for pH control and no sparingly soluble water treatment additives. However, it was found to be somewhat incompatible with a commercial organo-molybdate corrosion inhibitor and this type of interaction may limit the usefulness of the drag reducing agent.

Tests on the adverse effects of the drag reducing agent on heat transfer in heat exchangers are in progress and the results will be available shortly.

Large system tests on drag reducing agent performance in both chilled and hot water distribution systems are planned for the Spring of 1990; additional studies of ice slurry performance and behavior with and without drag reducing agents are in progress; economic assessment studies are also underway; and further new drag reducing agent development and corrosion inhibitor interaction studies are planned for the near future.

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HYDRAULIC CHARACTERISTICS OF ICE SLURRIES AND CHILLED WATER FLOWS

1.0 INTRODUCTION

The Canadian Advanced Heat Transmission Fluids Group (CAHTFG) is an informal consortium of some twenty Canadian government agencies, national and provincial scientific laboratories, universities and private companies with diverse scientific, technologic and commercial interests in district heating and cooling.

As a result of an initiative of Michael Wiggin of CANMET, this research and development group was formed in 1987 under the auspices of Energy, Mines and Resources Canada. Dr. John C. O'C. Young of the Department of Chemistry of Saint Mary's University was awarded a contract by the Science Branch of Supply and Services Canada (Serial No. 23440-6-9152/01-ST, issued on November 26, 1986 and amended on November 26, 1987) to manage and provide supporting services for the group; services which were to include working with Canadian industries on developing proposals to the International Energy Agency (IEA) for work to be done in Canada on the development of advanced fluids for energy transmission in heating and cooling systems.

In June 1988, the CAHTFG was awarded a research contract by the International Energy Agency (IEA) in support of its program proposal on "Influences of Ice Slurry Characteristics on Hydraulic Behavior" (Young, 1988a) The principal task components of this proposal were:

1.0 Construction of an ice slurry generating and storage system and instrumented flow loop fitted with pumps and heat exchangers for cool delivery;

2.0 Hydraulic and thermal testing of this system with ice-free chilled water containing water treatment additives (variables: pipe diameter, flow rate, temperature, pressure drop, heat transfer rate, heat exchange);

3.0 Similar tests on ice-free chilled water containing hydraulic drag reducing agents with and without water treatment additives;

4.0 Ice slurry studies of:

- 4.1 Effects of the presence of ice solids on pipeline pressure drop and heat exchange over ranges of ice solids loading and fluid flow rate;
- 4.2 Effects of hydraulic drag reducing additives on pressure drop and heat exchange over ranges of dosage, ice solids loading and fluid flow rate;
- 4.3 Effects of ice crystal size and shape on system hydraulics;
- 4.4 Effects of pipeline diameter on hydraulic behavior;

5.0 Scaleup studies and pilot plant proving tests under selected test conditions preparatory to full-scale demonstration.

This proposal was based on certain premises, one of which was that available technology would provide suitable drag reducing additives for our use at temperatures down to and somewhat below the normal freezing point of water. In the event this proved not to be the case, despite published claims to the contrary (Ohlendorf *et al.*, 1988). This finding necessitated a

major redirection of our efforts towards drag reducing additive development, somewhat at the expense of the later phases of the work we had intended to carry out under topic 5.0.

However, this final report under our current IEA contract represents only an interim presentation of our work on its subject, and we continue to be engaged in studies of i) new ice-making technologies and economics, ii) thermal and hydraulic behaviors of ice slurry and chilled water flows, iii) novel drag reducing agent formulations, iv) the economics of drag reducing agent manufacture and use, iv) interactions between drag reducing agents and conventional water treatment chemicals, and vi) pilot-scale testing and full-scale demonstration of the hydraulic behavior and thermal performance of ice slurry and drag reducing agent combinations.

Our report is presented under the following headings:

2.0 Drag Reducing Agent Development Studies (not listed in original research proposal (Young, 1988a)

3.0 Ice Slurry Flow Studies (Tasks 1.0, 2.0, 4.1, 4.3 and 4.4)

4.0 Drag Reducing Agent Flow Studies (Tasks 3.0)

5.0 Ice Slurry Plus Drag Reducing Agent Flow Studies (Task 4.2)

6.0 Scaleup Criteria, Pilot-Scale Testing and Full-Scale Demonstration (Task 5.0)

7.0 Summary, Conclusions and Future Work

8.0 References

9.0 Acknowlegments

2.0 DRAG REDUCING AGENT DEVELOPMENT STUDIES

2.1 BACKGROUND (Saint Mary's University)

The use of chemical additives as hydraulic drag reducing agents to increase liquid and slurry flows through pipelines has been widely studied, and comprehensive reviews of this work have been made by Hoyt (1972) and Shenoy (1984). However, these reviews make no reference to applications at temperatures appreciably below ambient, which are of interest for space cooling and other refrigerative systems employing chilled water and ice slurry flows. For such applications, the temperature range from -5 to +15 deg. C is of particular interest, and compatibility with polyhydric alcohols, corrosion inhibitors and other solutes may also be required.

More recently, Ohlendorf and his co-workers (1988) have recommended the use of n-dodecyl(lauryl)trimethylammonium salicylate (n-DTMASal) as a drag reducing agent for the temperature range 0-30 deg. C., but presented no experimental data to support this claim.

To test this claim, the Saint Mary's University (SMU) Research Group constructed a simple turbulent flow rheometer (Murray, 1989) and carried out preliminary drag reduction tests on 5-20 millimolar (mM) solutions of n-DTMASal in both water and 10% w/w ethylene glycol solution at temperatures from ambient to below 0 deg. C. The additive used was prepared from equimolar amounts of a commercial grade n-DTMA chloride (Arquad 12-50, Akzo

Chemicals Inc., Chicago, Ill., USA) and analytical grade sodium salicylate dissolved in the glycol solution. Salient results of this evaluation were as follows:

i) at 10 deg. C, a 20 mM solution of n-DTMASal showed 67% drag reduction at the highest wall shear stress tested (ca. 200 Pa), and similar performance was observed when the pH of the solution was raised from 6 to 12 by addition of solid caustic soda;

ii) supercooling occurred when a 20 mM solution of n-DMTASal was cooled to 2 deg. C and drag reduction was observed over several rheometer flow cycles before precipitation occurred and the drag reduction effect was lost; and

iii) 5 and 10 mM solution of n-DTMASal behaved similarly at 2 deg. C, and drag reduction also ceased when these solution were heated to 22 deg. C indicating that rod-to-sphere transformations had occurred.

Rod-to-sphere transformation was confirmed by measurements made in a continuous flow filter fluorometer adapted for use as a nephelometer (FIGURE 1). Light scattering was observed to be quite dependent on the fluid flow rate though the flowthrough cuvette at fluid temperatures below ca. 20 deg. C, but to be virtually independent of flow rate at higher temperatures.

These results indicate a useful temperature range of say 5 to 20 deg. C for n-DTMASal. At lower temperatures, transient supercooling was followed by precipitation of the additive and cessation of drag reduction. This behavior was reproducible upon heating to dissolve the precipitate and then recooling the solution (Young, 1988a, 1989a); and it indicates that the Krafft point of n-DTMASal, that is the temperature at which the solubility of its monomeric form reaches the critical micellar concentration (cmc) level, is close to 5 deg. C. A necessary but insufficient requirement for drag reducing capability is that the monomeric form of the additive be sufficiently soluble for its concentration in solution to reach the minimum level necessary for colloidal micelle formation, namely the critical micellar concentration, below the the desired operating temperature range. Another requirement is that the additive be capable of forming assymetric, "rod-like" micelles under the desired operating conditions (see, for example, Ohlendorf *et al.*, 1986).

In regard to higher temperature performance, 5 mM dosages of n-DTMASal provided significant drag reduction at 10 deg. C, but this addition level proved ineffective at temperatures above 20-22 deg. C. Flow nephelometric measurements indicated that this loss of drag reducing capability was attributable to a rodlike-to-spherical micellar transformation (Murray, 1989). Such transformations with temperature increase and an associated loss of drag reducing capability have been reported previously by Ohlendorf *et al.* (1986) for n-DTMASal and its n-tetradecyl (myristyl) and n-hexadecyl (cetyl) homologs. Unfortunately, these workers did not obtain micellar transformation concentration (mtc) data for n-DTMA(lauryl)Sal at temperatures below 35 deg. (FIGURE 2). However, an extrapolation of the given data suggests a micellar transformation temperature between 25 and 30 deg. C at the 5 mM dosage level (FIGURE 3: n-DTMASal contains 12 alkyl group carbon atoms), and near 20 deg. C for a 4 mM dosage (FIGURE 2). Given the uncertainty of this extrapolation procedure, these data are in reasonable agreement with our observations.

Thus, an unacceptably high Krafft point and a narrow temperature range over which rodlike micelle formation can be achieved at practical dosage levels, combine to eliminate n-DTMASal from contention as a drag reducing agent for ice slurry and chilled water applications.

Following this finding, the SMU Research Group embarked on a broadly-based effort to develop new drag reducing agents suitable for low temperature applications, and this activity became its principal effort within the contract period. Laboratory systems were constructed and test procedures were developed for studying various aspects of the behavior of colloidal micellar systems. This task is nearly complete, and procedures and equipment currently available include nuclear magnetic resonance evaluation of counterion effectiveness, nephelometric assessment of micellar shape, electrical conductometric determination of critical micellar concentration, solution density and low-shear viscocity measurement, a double syringe pump turbulent flow micro-rheometer for rapid screening of the hydraulic behavior of candidate micellar systems, and small-scale continuous and batch flow test units for predicting additive effectiveness under pipeline flow conditions. Details of this equipment and these experimental procedures are provided elsewhere (Murray, 1989). Towards the end of the contract period, this analytical capability was considerably enhanced by the purchase of a novel low-shear, falling pin viscometer (Park and Irvine, 1988) with supplementary contract funding provided by the International Energy Agency.

2.2 DRAG REDUCING AGENT SELECTION

2.2.1 Selection of the Surfactant Cation (Saint Mary's University)

Recognized hydraulic drag reducing agents of the type represented by n-DTMASal consists of two components, a positively charged n-alkyl trimethylammonium cationic surfactant and a negatively charged anionic counterion which aids micelle formation by reducing electrostatic repulsion between neighboring surfactant ions in the micellar structure. As indicated above (SECTION 1.1), the ability of these surface active compounds to act as drag reducing agents is associated with their capacity to form assymetric, rodlike colloidal micelles. Assymetric micelle formation is achieved only over the temperature range between the Krafft point and the micellar transformation temperature.

Each of the two additive constituents, the surfactant and the counterion, must be chosen to ensure that the Krafft point of the additive pair lies below the lower limit of the desired operating temperature range. This is requirement is relatively easy to meet for operating temperatures above ambient, but is not met by any of the additives thusfar recommended in the literature when employed at temperatures close to and below the normal freezing point of water. For this below-ambient temperature range, the reduced solubility of the monomeric form of the additive greatly restricts the choice of suitable candidate compounds.

As is illustrated in FIGURE 4, the Krafft point of a given additive system is defined by the intersection between its monomer solubility vs. temperature relationship and the relationship between solution temperature and the minimum additive concentration necessary for micelle formation to occur (the cmc value). The Krafft point may be reduced by modifying the composition of the additive so as to raise its solubility and or reduce its cmc value.

Homologs of n-dodecyl (lauryl) trimethylammonium salicylate (n-DTMASal) which contain longer alkyl chains are more amphiphilic, and therefore exhibit stronger micelle forming tendencies and hence lower cmc values. Data obtained by Ohlendorf *et al.* (1986) illustrate this trend for lauryl, myristyl and hexadecyl trimethylammonium salicylates, which showed cmc values of ca. 2.5, 0.5 and 0.1 mM respectively at 20 deg. C (FIGURE 2). However, these two higher homologs of n-DTMA(lauryl)Sal are much less soluble in water, and our observations indicate that the effect of lower solubility of elevating the Krafft point more than compensates for the effect of lower cmc value of depressing it. Thus, within this homologous series, the net effect is an increase in Krafft point with increase in alkyl chain length.

This effect is illustrated by results of our studies of the drag reducing capabilities of 20 mM solutions of n-hexadecyl (cetyl) trimethylammonium salicylate (CTMASal), which showed precipitation and neglible drag reduction at 10 deg. C, indicating a Krafft point appreciably above 10 deg. C; whereas a Krafft point near 5 deg. C was observed for n-DTMASal.

These CTMASal solutions also provided 65-70% drag reduction at 35°C (the maximum temperature examined), indicating an upward shift in the rod-to-sphere transformation temperature as compared with that observed for n-DTMASal, which showed negligible drag reduction above 20 ^{o}C at the same 5 mM dosage level.

The useful operating temperature range of a drag reducing additive is bounded by its Krafft point and by its rod-to-sphere transformation temperature which is depends both on dosage level and the applied wall shear stress. Under "all else equal conditions" of dosage and stress, it appears that this useful operating temperature range shifts upward with alkyl chain length for n-DTMASAl and its homologs, and this type of trend with increasing amphiphilicity has also been reported by Icking *et al.* (1989) for members of a homologous series of alkyl trimethylammonium naphthoates.

As indicated above, the effective operating temperature range of the dodecyl member of the n-alkyl TMASal series is too high to be useful for ice slurry-based cooling units and is questionable for chilled water systems, and use of the decyl member was therefore considered.

Mukerjee and Mysels (1971) have reported the following cmc data for n-alkyl trimethylammonium chlorides :

Surfactant Species	CMC (mM) at 25 deg. C
C_{10} trimethylammonium chloride	61.1
C_{12} trimethylammonium chloride	20.3
C_{14} trimethylammonium chloride	4.5
C_{16} trimethylammonium chloride	1.3
C_{18} trimethylammonium chloride	0.4

The data reported by Ohlendorf *et al.* (1986) indicate cmc values of ca. 2.5, 0.5 and 0.1 mM respectively at 25 deg. C for the corresponding salicylates of the three middle members of this tabulation (FIGURE 2), indicating the marked superiority of salicylate over chloride as an aid to micellation. These data correspond to 8-10 fold reduction in cmc value between these two counterions, and therefore suggest a cmc value of say (61/10=) 6 mM for n-decyl trimethylammonium salicylate.

Even if a cmc value as high as 6 mM were economically acceptable for drag reduction applications, a cross-plot of selected data from FIGURE 2, which is shown in FIGURE 3, reveals a further problem with the use of n-decyl trimethylammonium salicylate. This plot indicates a rod-to-sphere micellar transition temperature close to ambient at the 6 mM dosage level. Such a micellar transformation could therefore occur even in low temperature systems during downtime periods, and its appears to be only slowly and possibly incompletely reversible upon recooling. (We plan further studies of reversibility of the rod-to-sphere and sphere-to-rod processes). This transformation should not occur with n-DTMASal, for which the transition temperature is almost 40 deg. C (FIGURE 3).

It therefore appeared that the 14-carbon chain length was optimum for low temperature applications. Initial results of a continuing study of the effects of unsaturation in the dodecyl group and ethoxylation in the other groups attached to the quaternary nitrogen show either unchanged or inferior drag reduction results compared with those obtained with n-DTMASal. Although the best of the compounds tested, it was clear that n-DTMASal was marginal for chilled water applications and unsuitable for use at the operating temperature range of ice slurry systems.

Given these findings, a decision was made to accept n-dodecyl TMA cation as the reference surfactant component for low temperature applications, and to attempt to depress its useful operating range somewhat by searching for a more effective counterion than salicylate to aid micelle formation.

In quantitative terms, the aim of this search became to find a counterion for the n-dodecyl trimethylammonium cation which would reduce the Krafft point of the additive system below -5 deg. C, while maintaining a rod-to-sphere transition temperature of at least 25 deg. C at a dosage level of 5 mM.

2.2.2 Selection of the Counter-Anion (Saint Mary's University)

As part of this project, the SMU Research Group made an extensive study of the role of the counterion in micelle creation (Murray, 1989), which included an experimental investigation of relationships between the molecular structures of various benzene-derived anions and their efficacy as promoters of micelle formation. More recently, this study was extended to include naphthylene-based counterions (Cleary *et al.*, 1989). The behavioral model developed in the course of these studies suggests that, to be effective as a counterion, a candidate compound should be unbranched and amphiphilic and should possess a structure such that its negative charge is highly dispersed over its head group. An unbranched hydrophobic tail group attached to a hydrophilic head group favors alignment of the tail group within the body of the micelle, while charge dispersion facilitates neutralization of the positive charges on neighboring surfactant cations at the surface of the micelle.

Micelle formation, and particularly rodlike micelle formation, is accompanied by large increases in solution viscocity which are associated with long-range solution order effects.

Totten *et al.* (1986) have reported the following kinematic viscocity data for 200mM solutions of myristyl TMA surfactant with various aryl carboxylate and sulfonate counterions:

Aromatic Counterion	Viscocity (cSt)
4-hydroxybenzoate	1
benzoate	19
2,4-dihydroxybenzoate	84
2-mercaptobenzoate	111
2-aminobenzoate	131
2,5-dihydroxybenzoate	388
2-hydroxybenzoate (salicylate)	6275
2,6-dihydroxybenzoate	coacervate
4-hydroxybenzenesulfonate	1
benzenesulfonate	287
m-benzenedisulfonate	594
2-aminobenzenesulfonate	4168
2-hydroxybenzenesulfonate	precipitate

Viscocity increases down these benzoate and sulfonate series are associated with increasing solution order in the progression i) single ions, ii) ion pairs, iii) spherical micelles, and iv) rod shaped or lamellar micelles, followed by coacervation and precipitation.

Relating to counterion design, these data provide a number of informative trends and comparisons:

i) 4-hydroxybenzoate (1 cSt) and 4-hydroxy benzenesulfonate (1) lack amphiphilicity because they contain hydrophilic substituents in both the "1" and "4" positions. Addition of the para hydroxyl group makes them inferior to benzoate (19) and benzenesulfonate (287) respectively;

ii) benzoate (19) is weakly amphilic;

iii) 2,4-dihydroxybenzoate (84) lacks amphiphilicity because it also contains hydrophilic substituents in the "1" and "4" positions. However, as compared with 4-hydroxybenzoate (1), the additional hydroxyl group in the ortho position relative to the carboxyl head group ensures a measure of delocalization of the negative charge on the carboxyl group;

iv) 2,5-dihydroxybenzoate (388) is somewhat more amphiphilic than 2,4-dihydroxybenzoate (84) and, as indicated, this shifting of a hydroxyl group from the "4" to "5" position has an appreciable effect on viscocity;

v) 2-hydroxybenzoate or salicylate (6275) may be envisaged as 2,5-dihydroxybenzoate (388) with a hydroxyl group removed from the "5" position to produce a more amphiphilic structure;

vi) 2,6-dihydroxybenzoate (coacervate) may be envisaged as 2,5-dihydroxy benzoate (388) with a hydroxyl group moved from the "5" to "6" position to produce a more amphiphilic structure and further delocalization of the negative charge on the carboxyl group;

vii) 2,6-dihydroxybenzoate (coacervate) may also be envisaged as 2-hydroxy benzoate or salicylate (6275) with a second hydroxyl group substituted in the other position adjacent to the carboxyl group to enhance both charge delocalization and amphiphilic character. Salicylate is the counterion most commonly used by previous workers, and it therefore serves as a useful benchmark;

viii) the series 2-hydroxybenzoate (6275), 2-aminobenzoate (131) and 2-mercaptobenzoate (111) and benzoate (19) indicates that hydroxyl is a more effective electron-withdrawing substituent than amino or mercapto groups; and

ix) the pairs benzoate (19) versus benzenesulfonate (287), 2-aminobenzoate (131) versus 2-aminobenzenesulfonate (4186) and 2-hydroxybenzoate (6275) versus 2-hydroxybenzene-sulfonate (precipitate) all suggest that sulfonate may be a more effective counterion than carboxylate.

These data clearly suggest the importance of amphiphilic character and particularly of head group charge delocalization to solution viscocity and therefore presumably to micellation and drag reducing capability.

The desired improvement on the counterion performance of salicylate might therefore be achieved by i) insertion of a short n-alkyl group in the "3", "4" (preferably) or "5" position to the carboxyl group to improve amphiphilicity and ii) insertion of a second hydroxyl group adjacent to the carboxyl group to improve charge delocalization.

The first of these two measures has been suggested elsewhere by Ohlendorf *et al.* (1986) whose relevant patent claims refer to benzoate and sulfonate counterions with "a hydrogen or hydroxyl in position 2 or 3" and a " C_1-C_5-alkyl, C_2-C_5-alkenyl or C_1-C_5-alkoxy in position 3, 4, 5 or 6". A number of the structures envisaged in this claim would appear to be less effective, but benzoate or sulfonate structures with a hydroxyl group in the "2" position and an n-alkyl group in the "3", "4' or "5" position appear promising. These workers presented supporting data indicating that salicylate, 3-methylsalicylate, 4-methylsalicylate, 4-ethoxysalicylate and 4-methyl-3-hydroxybenzoate were similarly effective as counterions with cetyl trimethylammonium cation at ambient temperature.

No literature references were found to the use of 2,6-dihydroxy benzoates and sulfonates.

It would have been interesting and informative to examine the drag reducing performance of various such dihydroxy compounds with and without para-positioned hydrophobic substituents, but time constraints did not permit the development of appropriate synthesis procedures. However, screening tests were made on the following commercially available compounds amongst others (Cleary *et al.*, 1989):

Counterion	deg. C	% Drag Reduction	Viscocity (cp)
3-methylsalicylate	10	66	2.29
2,6-dihydroxybenzoate	10	65	5.07
2,6-dihydroxybenzoate	2	64	6.79
salicylate	10	64	2.40
4-methylsalicylate	2	63	2.76
1,4-dihydroxy-2-naphthoate	7	54	4.64
2-hydroxy-1-naphthoate	7	48	2.16
4-methylbenzoate	2	9	1.98
4-methylsulfonate	10	4	1.81
4-hydroxybenzoate	5	- 2	2.32
3-hydroxy-2-naphthoate	2	- 2	2.88

For economic reasons, a low critical micellar concentration (cmc) value is a desirable attribute of a drag reducing agent. Because it was more convenient to make measurements at room temperature, which is well within the operating range of n-tetradecyl (myristyl) trimethylammonium salicylate, the n-TTMA surfactant was used in place of n-DTMA in the following comparitive evaluation of counterion effects on cmc value (Cleary *et al.*, 1989):

Counterion	cmc Value (mM)
2,6-dihydroxybenzoate	0.25
4-methylsalicylate	0.40
salicylate	0.62
4-hydroxybenzoate	1.91
3-hydroxy-2-naphthoate	4.10

The counterions 2,6-dihydroxybenzoate and 4-methylsalicylate, which showed low cmc values and good low temperature drag reducing performance in these screening tests, were selected for more detailed evaluation in the dual-tube rheometer shown in FIGURE 5. This test unit provides side-by-side comparisons of the flow rates obtainable with and without the drag reducing agent under identical conditions of tube geometry (1.0 m length, 2.2 mm diameter), fluid temperature and wall shear. All tests were made in the presence of 10% w/w ethylene glycol using the formulation Arquad 12-50 (Akzo Chemicals Inc.) as the source of n-DTMA ion.

Representative sets of comparitive flow data for 10 mM dosages of n-DTMA with equimolar amounts of 2,6-dihydroxybenzoate and 4-methyl salicylate at -2 deg. C are shown in FIGURE 6. Given that an applied pressure gradient of 1 kPa/m is equivalent to a wall shear stress of 1.1 Pa for the experimental tube diameter of 2.2 mm, these data indicate that 4-methyl salicylate induced somewhat higher flow rates for wall shear stress levels below 300 Pa, possibly because its viscocity in 10 mM solution is appreciably lower than that of 2,6-dihydroxybenzoate (see viscocity data reported above). Presumably because it is a more effective counterion and binds rodlike micelles together more tightly, 2,6-dihydroxybenzoate was more effective than 4-methylbenzoate at wall shear stresses above 300 Pa.

Effects of dosage level are shown in FIGURES 7 and 8. As compared with the glycol solution flow, the 2,6-dihydroxybenzoate additive produced the following percentage flow rate increases:

Shear Stress (Pa)	Gradient (kPa/m)	5 mM Dosage	10 mM Dosage
100	90	33%	21%
200	180	82%	81%
300	270	94%	105%
400	360	66%	90%

These data suggest that, even for the lower dosage level, the critical wall shear level at which the drag reduction ceases is above 400 Pa at the test temperature of 2 deg. C. The somewhat inferior performance observed at low shear stress levels for the higher (10mM) dosage is attributable to the substantial effect of the presence of the additive on the viscocity of the solution. Viscocity effects are of lesser significance at the higher shear stress levels associated with fully developed turbulent flow, in which the higher dosage is more effective presumably because it contributes to the formation of more shear-resistant rodlike micelles. Effects of dosage level on the Darcy-Weisbach friction factor are illustrated in FIGURE 9. Relationships between solvent Reynolds Number and friction factor with and without additive are shown in FIGURE 10.

Rose (1985) has found that a greater than equimolar addition of the counterion substantially improved the drag reducing performance of cetyl trimethylammonium salicylate. Effects of solution temperature and counterion concentration on the flow rate enhancing capability of our n-DTMA 2,6-DHB additive system for a wall shear stress of ca. 250 Pa are shown in FIGURE 11. At low temperatures, raising the counterion concentration by 50% had a slightly adverse influence, presumably because of an associated increase in viscocity. At higher temperatures, the additional counterion concentration apparently stabilized micelles against thermal disruption and raised the temperature at which drag reduction ceased from 30 to over 40 deg. C. The additional counterions would presumably also increase micelle resistance to shear stress at constant temperature.

Based on the foregoing study results, n-DTMA 2,6-DHB was selected as our first proposed additive for low temperature applications, and samples were delivered to the Dearborn Chemical Company Ltd. for compatibility testing with corrosion inhibitors and water treatment chemicals (SECTION 2.2.3); and to the Low Temperature Laboratory of the National Research Council of Canada for continuous flow testing and durability assessment in chilled water (SECTION 4.0) and ice slurries (SECTION 5.0).

2.2.3 Compatibility With Other Additives (Saint Mary's University and the Dearborn Chemical Company Ltd.)

For corrosion inhibition in low temperature water-based systems, the Dearborn Chemical Company recommended use of its formulation "Dearborn 546", which it describes as " . . . a highly buffered, molybdate based corrosion inhibitor for use in multi-metal closed recirculating water systems that are subjected to air ingress contamination and/or trace acid contaminants. . . ", with the claim that "Complete protection is provided against corrosion for

ferrous and non-ferrous metals in both chilled and hot water heating systems" (Dearborn Bulletin 22-546).

Using this product and also a competitive silicate-based corrosion inhibitor (Betz Inc. Product No. 545), Dearborn carried out a series of corrosion inhibition tests in the presence of three drag reducing formulations prepared by the SMU Group as 1 M stock solutions with 50% excess counterion, namely n-DTMA 2,6-DHB, n-DTMASal and n-dodecyl pyridinium salicylate (n-DPyrSal).

Details of the corrosion test procedure used and the results obtained have been reported elsewhere by Thompson (1988, 1989(a), 1989 (b)). Corrosion rate data obtained from the first test series (Thompson, 1988) included the following results for inhibitor dosages of 3000 mg/l of Dearborn 546 (equivalent to 78 mg/l of Mo) or 500 mg/l of Betz 545 (equivalent to 30 mg/l SiO_2) which were obtained for stirred sample solutions at room temperature and pH values near 8:

Additive	Dosage*	Treatment	Fe Corrosion	Cu Corrosion
n-DTMASal	none	D546	0.44 mm/yr	0.01 mm/yr
		low	0.19	0.05
		inter	0.26	0.04
		high	0.37	0.09
n-DTMASal	none	B545	0.90 mm/yr	0.00 mm/yr
		low	0.81	0.02
		inter	0.78	0.05
		high	0.37	0.07
n-DPyrSal	none	D546	0.44 mm/yr	0.01 mm/yr
		low	0.17	0.02
		inter	0.17	0.05
		high	0.33	0.07
n-DPyrSal	none	B545	0.90 mm/yr	0.00 mm/yr
		low	0.52	0.04
		inter	0.35	0.03
		high	0.42	0.06

(* "Low", "Inter(mediate)" and "High" refer to 1, 2 and 5 mM dosages)

These corrosion rates are reasonable to low, judged against a benchmark rate of 1 mm per year for ferrous corrosion. The data also suggest that the drag reducing agents have some corrosion inhibiting capability in their own right. However, crevice corrosion of the steel coupons was reported, and coloration of and floc formation in the test solutions were also observed. Also, progressive loss of residual dissolved molybdenum or silicate were observed with increase of the drag reducing agent dosage.

Thompson (1989(a)) attributed the formation of flocs in these test solutions to a reaction between the cationic drag reducing agents and an anionic polymer component of the corrosion inhibitor formulations. Following the removal of this component, the following test data were obtained for this modified version of the Dearborn 546 additive:

Additive	Dosage	Treatment	Fe Corrosion	Dissolved Fe	Residual Mo
n-DTMASal	none	D546Mod	0.15 mm/yr	1.17 mg/l*	77 mg/l
	low		0.08	0.06	73
	inter		0.17	1.25	63
	high		0.45	3.10	44

(* expressed as iron (III) oxide Fe_2O_3).

Floc-free, colored solutions were obtained in all cases, confirming that a reaction with the polymer component was responsible for floc formation and that coloration was attributable to the drag reducing agent. As in the previous test series, corrosion inhibition was observed for the "low" drag reducing agent dosage but corrosion rates rose progressively as this dosage was raised. Again, corrosion rates were judged to to be low or reasonable in all cases.

Corresponding tests on n-DTMA 2,6DHB with the modified corrosion inhibitor formulation yielded the following results (Thompson, 1989(b)):

Additive	Dosage	Treatment	Fe Corrosion	Dissolved Fe	Residual Mo
DTMA2,6DHB	none	D546Mod	0.98 mm/yr	0.45 mg/l*	60 mg/l
	low		0.53	0.16	48
	inter		1.72	1.80	28
	high		2.49	3.60	18

(* expressed as iron (III) oxide Fe_2O_3).

Sample solutions were floc-free but highly colored and steel test coupons showed light to heavy crevice corrosion. These results again indicate an adverse reaction between the corrosion inhibitor and the drag reducing agent, resulting in this case in higher corrosion rates than would be acceptable in an operating system.

The Dearborn Chemical Company is continuing its effort to find an inhibitor system which is compatible with cationic drag reducing agents.

The SMU group tested the effect of the (unmodified) Dearborn 546 corrosion inhibiting formulation on the behavior and performance of a 10 mM dosage of n-DTMA 2,6 DHB in 10% w/w ethylene glycol solution. The formation of a precipitate was observed upon adding 0.3% w/w of this corrosion inhibitor. From observed differences in the coloration of a series of solutions containing this concentration of corrosion inhibitor with varying amounts of the drag reducing agent, it appeared that one third to one half of the latter was being precipitated by the corrosion inhibitor. Consistent with this observation, approximately half of the flow enhancing capability of the drag reducing agent was lost. Furthermore, essentially normal flow enhancement for a 10 mM dosage of the drag reducing agent was achieved by raising the dosage of 15 mM.

However, while effects of the presence of the corrosion inhibitor on the performance of the drag reducing agent may be overcome by raising the dosage of the latter, precipitation by the drag reducing agent of the corrosion inhibitor might be expected to impair its inhibiting capability, and this proved to be the case (Thompson, 1989(b)).

Rather than employing chemical corrosion inhibitors, some systems use "pure" deionized, deaerated water with sodium hydroxide addition for pH control. These conditions are more favorable to the use of our proposed drag reducing additive systems, since interactions with inhibitors are eliminated and the SMU Group has found no significant effect of pH value on performance at least over the range of 6 to 10.

Continuous flow testing of the n-DTMA 2,6DHB drag reducing agent without corrosion inhibitors was carried out in the Low Temperature Laboratory of the National Research Council of Canada, both in chilled water (SECTION 4.0) and ice slurries (SECTION 5.0).

3.0 ICE SLURRY FLOW STUDIES

3.1 BACKGROUND LITERATURE REVIEW (Saint Mary's University)

Pumpable ice slurries offer considerable advantages over chilled water as a medium for storing and delivering "cold" for space cooling applications. Compared with chilled water which can provide 20-40 megajoules per cubic metre of water for an operating temperature range of 5-10 deg. C, a 25% solids ice slurry can provide 80 megajoule per cubic metre with greater thermal delivery efficiency and neglecting any sensible heat (cold) contribution.

However, ice is not the ideal latent heat (cold) storage medium because its melting point of 0 deg. C (or lower in the presence of solutes) is lower than optimum for space cooling requirements. Lower operating temperatures increase condensation (dehumidification) problems and storage heat gains, and adversely affect the thermodynamic efficiency of the refrigerating system. This last effect is indicated by the the substantial effect of evaporator temperature on the efficiency of a residential vapor compression heat pump (Allen and Kani. 1985):

Evaporator Temperature (deg. C)	Heat Output Rate (kW)	Heating COP*
-12	3.7	2.3
0	4.9	3.3
5	5.5	3.6
10	6.9	4.1
25	8.3	4.4

(* COP(Cooling) = COP(Heating) - 1)

These data indicate that, for example, the coefficient of performance for cooling rises from 1.3 at -12 deg. C to 2.6 at +5 deg. C, corresponding to a doubling of cold delivery per unit electrical input over this temperature range.

According to a panel of experts convened by EPRI to consider new energy storage opportunities (Electric Power Research Institute, 1983),

> . . . the freezing (melting?) temperature of ice, 32 deg. F (0 deg. C), is much lower than desirable for use with conventional air conditioning equipment . . . A storage medium with a freezing point of 50 deg. F (10 deg. C) plus or minus a few degrees would be ideal.

Elsewhere, Ternes (1985) suggests that cool should be stored between 5 and 13 deg. C for optimum system performance, and notes that the efficiency and capacity of most heat pump and air conditioning units decline rapidly as their evaporator temperatures fall below 5 deg. C.

Ternes (1985) and others have proposed the use of ice clathrates to provide perhaps 80% of the latent heat of fusion of ice at say 10 deg. C. However, this type of "warm ice" process is at an early stage of development and still requires major efforts in a number of areas, including direct-contact heat transfer and oil-less compressor design (Knodel, 1988). In the meantime, transportable and storable ice slurries are a potentially valuable "bridging technology".

Regarding such pumpable ice slurries, a few studies have been reported of the effects of ice particles on hydraulic drag in pipelines. For ca. 15% v/v suspensions of very coarse ice particles of mean diameter 1.2 cm in water flowing through a horizontal pipe of diameter 0.10 m, Sellgren (1986) found friction factor values to be similar to those for solids-free water at velocities above 1-1.5 m/s, but to be appreciably greater than those of water at lower velocities. This behavior was attributed to additional frictional drag caused by ice separation in the top half of the pipe cross-section at low water velocities.

The opposite trend towards lower drag of ice slurries than for solids-free water has been reported by Knodel (1988) who observed that ice slurries containing ca. 10% v/v solids showed 25-30% less drag than water flowing through the same 0.10 m diameter pipe over the same velocity range of 1.5-3 m/s. The proprietary direct-contact refrigeration unit (CBI Inc., Plainfield, Illinois) used in this study produced relatively coarse ice particles with typical diameters of 0.3 to 0.6 cm. These tests were carried out in transparent pipe and the following flow behavior was reported:

> At low solids loading, ice fractions below 2%, the ice particles which range from 1/8 inch to 1/4 inch (0.3 to 0.6 cm) in diameter move with a random motion throughout the the cross section of the flow resembling a classical turbulent flow pattern. As the concentration is increased, the ice crystals form small clusters within the flow 1/2 inch to 3/4 inch (1.2 to 1.9 cm) in diameter. The motion of these clusters is more restrained. (It is important to note, however, that these clusters are easily broken up in regions of high shear such as pipe bends outside of the test section). Further increases in concentration produce larger clusters and less motion. At ice fractions of approximately 8 to 10%, the ice slurry takes on a fully developed plug flow pattern . . . The mechanism for the associated drag reduction is dampening of turbulence in the (central) plug region of the flow by the solid particles.

Turbulence dampening behavior has been reported for the hydraulic transportation of various minerals (Thomas, 1962), sand and clay mixtures (Durand, 1952) and wood pulp (Stepanoff, 1964). However, if the solids loading is progressively raised, hydraulic drag must surely eventually increase as the central plug flow zone expands outwards to the pipe wall.

Combining the foregoing observations, wall friction might be expected to vary little with ice solids loading at "low" concentrations, (possibly) to fall at "intermediate" concentrations and (certainly) to rise at "high" concentrations.

These studies suggest that, over considerable ranges of fluid velocity and ice solids loading, ice particles might act as "flow straightening" additives which extend the region of subturbulent flow conditions to higher velocities and thereby reduce pipe friction losses below those experienced with water alone under otherwise similar hydraulic conditions.

Clearly, much remains to be learned about the effects of particle size, shape and loading on the hydraulic behavior of ice slurries.

3.2 HYDRAULIC AND THERMAL STUDIES OF ICE SLURRY FLOWS (National Research Council of Canada)

To investigate the hydraulic and thermal properties of flowing ice slurries, a flow test loop was constructed in the Low Temperature Laboratory of the National Research Council of Canada (FIGURE 12) (Larkin, 1989).

In this system, ice is produced at a peak rate of ca. 100 kg/hr in the proprietary wiped surface heat exchanger of a Sunwell Model IG2-WS6 vapor compression refrigeration unit (manufactured by project participant Sunwell Engineering Company, Mississauga, Ontario). In this exchanger, high transfer rates are achieved by causing ice formation to occur in the bulk of the 6% glycol solution medium rather than on the heat exchange surface. The mean particle size of the freshly produced ice slurry is quite small, averaging 0.005-0.010 inch or approximately 0.1 - 0.2 mm.

The ice slurry produced is delivered to a stirred storage tank, from which it can be pumped through the flow test loop or through the heat exchanger test section. The flow loop contains 15 m long test sections of 25 mm and 50 mm internal diameter, fabricated from smooth copper pipe and fitted with pressure taps, thermocouples and flow meters. Instrumentation available for data gathering and reduction include a dedicated microcomputer, analog-to-digital signal converter, data logger, data processor and plotter.

The system includes two heat exchangers; (i) a small plate exchanger made up of 14 plates, each 7 cm by 18 cm, with a separation of 2.1 mm, and (ii) a shell-and-tube exchanger with sixteen 21 mm i.d. by 0.91 m long tubes connected in series and an unbaffled, single-pass shell side. The heat source for heat exchanger testing is a tank with a capacity of 11,000 litres of water. A turbine flow meter is used to measure flow on the water side of the heat exchangers.

The ice fraction in the slurry is measured by electrically melting the ice in a sample collected in a calorimeter (FIGURE 12) at the return outlet to the storage tank.

The system was originally fitted with a magnetic flow meter, which was expected to perform satisfactorily with ice slurries. However, when compared with flow weight measurements, its calibration was found to vary with ice solids concentration and Reynolds Number. A paddle flow meter and a turbine flow meter were tested but these instruments did not function when the ice fraction exceeded 10%. An ultrasonic flow meter was also tested and found to be unsatisfactory for ice slurry measurement.

Preliminary tests were run with water to check out the instrumentation, and friction factors obtained were found to be in agreement with literature values. Heat balances over the

heat exchangers were obtained within ± 2%. In subsequent tests, when the presence of ice prevented measurement of the heat flux on the slurry side, heat fluxes were calculated from the water side data.

The usual test procedure was to set the desired flow rate, and to start up the ice-making unit with the glycol "brine" solution temperature slightly above its freezing point. The test run then took place with a progressively increasing ice solids concentration.

A typical data set for this type of freezing test on the 25 mm diameter pipe is shown in FIGURE 13, and indicate a progressive decrease in flow velocity during the test as the overall flow resistance of the system increased. Similar relationships were obtained for flow velocities from 1.5 to 5 m/s in the 25 mm diameter section, and from 1.5 to 3.0 m/s in the 50 mm diameter section.

The f factor data shown in FIGURE 14 are corrected to a constant slurry flow velocity of 3 m/s through the 50 mm pipe. The f factor is the ratio of the observed friction factor for the slurry to that of the solids-free glycol solution at the same velocity, and at a viscocity value equal to that of the residual unfrozen concentrated glycol solution in the ice slurry. These data show that the f value remained essentially equal to that of the (concentrated) solids-free glycol solution for ice solids concentrations up to 30%, but rose sharply at higher solids concentrations. The data given in FIGURE 15 show that this critical solids concentration increases both with velocity and pipe diameter.

Two types of ice melting trial were carried out on the plate heat exchanger. Either (i) the ice making unit was started up and and the test initiated with chilled water flowing through the exchanger, or (ii) an inventory of ice was built up and the test initiated on ice slurry. Results of a typical test in the first "freezing run" category are shown in FIGURE 16, in which heat transfer efficiency is expressed as the overall heat exchange rate per unit (log-mean) temperature difference between the ice slurry and hot water flows. (To obtain overall heat transfer coefficients per square metre of exchange surface, the given values should be divided by the effective exchanger area, namely 0.184 m^2). The indicated slurry temperatures and ice slurry solids concentration levels were measured at the exchanger outlet, and the temperature discontinuity near 80 minutes corresponded to the first appearance of of ice solids in the exchanger effluent. The hot water side flow was maintained constant at 17 kg/min, but the slurry side flow rate varied somewhat through the run from an initial value of 9 kg/min down to slightly above 5 kg/min.

These data show a decline in the heat transfer coefficient and pressure drop across the exchanger throughout the test run. However , when the data are corrected to a constant mass flow rate of 9 kg/min, the heat transfer coefficient is seen to fall over the initial cooling period (FIGURE 17), during which the viscocity of the chilled water flow increases progressively, and then rises somewhat with the appearance of ice solids which might be expected to reduce the slurry side film resistance.

The data shown in FIGURE 18 were obtained from the second type of test and appear to be contradictory in that they indicate an increase in heat transfer rate coincident with the disappearance of ice solids. However, in this case, it is plausible that any adverse effect on heat transfer resulting from solids disppearance is more than offset by the favorable effect of the accompanying reduction in fluid viscocity as the temperature rose.

Given that the data in FIGURE 17 show that the heat transfer coefficient rose with increase in ice solids concentration in face of a (slight) decline in temperature, it seems clear that the presence of ice solids does improve heat transfer under isothermal conditions.

Typical test results for ice slurry flow through the tube side of the shell-and-tube heat exchanger, corrected to constant flow rate, are shown in FIGURE 19. Although less clearcut than in the case of the plate heat exchanger, these data also suggest that the heat transfer coefficient fell slightly over the cooling period before the start of ice formation and then rose slightly after ice formation began.

Finally under this topic heading, the question of effects of ice solids particle size on hydraulic behavior was briefly addressed. Ice slurries freshly produced by the ice-making unit have a mean particle diameter of ca. 0.1 mm (FIGURE 20). The ice particles were not completely round, but neither did they have sharp edges or corners.

In storage without additional refrigeration, considerable particle growth was observed despite the fact that the ice mass as a whole was slowly melting. The mean particle size increased to ca. 0.2 mm after one day and to 0.5 mm after three days, corresponding to a greater than 100-fold increase in mean particle volume over the three day storage period (FIGURE 20).

Some adverse effect on heat exchange efficiency might be expected to result from the loss of interfacial area caused by particle growth in storage. In this event, heat transfer coefficients obtained from melting runs, which involve the use of stored ice, might be expected to be lower than those observed in freezing runs at the same slurry flow rate and solids content. However, a comparison between a melting run and several freezing runs at the same solids concentration level provide no evidence of such an effect (FIGURE 21).

Furthermore when the heat transfer data for a given run are adjusted to a constant flow velocity, as for example in FIGURES 17, 18 and 19, a doubling or tripling of the solids concentration is shown to produce only a small effect on heat transfer rates. In view of the large changes in interfacial area associated with such increases in solids concentration, it is apparent that heat transfer rates are insensitive to interfacial area, indicating, as might be expected, that most of the slurry side thermal resistance lies at the pipe wall rather than at the ice particle-liquid interface.

In conclusion, it appears that the hydraulic and thermal behavior of the fine ice slurries produced in the experimental system will be essentially as that of the carrier liquid at the same temperature and ethylene glycol concentration, at least up to 25-30% solids for practical line diameters and operating velocities (FIGURE 15).

4.0 DRAG REDUCING AGENT FLOW STUDIES (National Research Council)

Because of time constraints, planned studies of the performance of the selected drag reducing agent in (ice-free) chilled water were deferred.

5.0 ICE SLURRY PLUS DRAG REDUCING AGENT FLOW STUDIES (NRC)

Studies of the performance of the selected drag reducing agent in ice slurries are currently in progress. To speed up these tests, a procedure for continuous measurement of the ice solids concentration has been developed. A small stream of the slurry is diverted through the plate heat exchanger, in which complete melting is achieved. The ice solids concentration is then determined from a heat balance over the heat exchanger. A comparison between results obtained by this procedure and the calorimetric method used previously is shown in FIGURE 22.

Initial results show a 60% reduction in pressure drop across the 25 mm diameter pipe section upon addition of a 10 mM dosage of n-DTMA 2,6-DHB drag reducing agent (FIGURE 23). The pressure drop reduction would have been greater, had it not caused a 20% increase in the slurry flow rate through the pipe.

Addition of the drag reducing agent caused an unexplained and apparently temporary three-fold increase in the pressure drop across the sidestream heat exchanger (FIGURE 23). Assuming the heat exchanger to be operating above the critical wall shear stress level of the drag reducing agent, the additive would have no beneficial influence on drag and would in fact be expected to cause some increase in pressure drop due to its effect of raising the viscocity of the slurry flow. The exchanger pressure drop declined somewhat as the ice solids fraction and hence the solution viscocity declined (FIGURE 23). However, the magnitude of the observed changes in the heat exchanger pressure drop appear too large to be attributable to viscocity effects alone, and additional tests are planned to examine effects of the drag reducing agent on drag through the exchanger and also on its heat transfer performance.

6.0 SCALEUP CRITERIA, PILOT TESTING AND FULL-SCALE DEMONSTRATION

Spot tests carried out to date suggest that constant wall shear may be an appropriate basis for scaling up laboratory test data on drag reducing performance to full-scale application.

Development of scaleup criteria will be a major component of the experimental plan for a large-scale test of drag reducing agent behavior and performance in a chilled water-based, university space cooling plant which the Canadian Advanced Heat Transmission Fluids Research Group is planning to carry out in the Spring of 1990. The CAHTFRG is also in negotiation to carry out an experimental study of drag reducing agent performance in a hot water-based district heating system.

Also towards full-scale demonstration and dissemination, the CAHTFRG is currently i) making a computer model assessment of the economics of ice slurry and drag reducing agent use; ii) developing a novel glycol-free bulk freezing ice-making process; iv) discussing drag reducing agent manufacture with several chemical manufacturers; and v) planning a service manual on the use of ice slurries and drag reducing agents.

7.0 SUMMARY, CONCLUSIONS AND FUTURE WORK

Considerable progress has been made towards the development of drag reducing agents suitable for low temperature applications. However, further work is warranted to improve additive performance, stability and compatibility with commercial corrosion inhibitors.

The results of initial test work on the use of pumpable ice slurries are promising, but much remains to be learned about their heat exchange performance, behavior in storage and interactions with drag reducing agents.

Work on drag reducing agent development and pumpable ice slurry performance evaluation are continuing at Saint Mary's University and the National Research Council of Canada respectively, and the CAHTFRG is currently planning activities in all of the activity areas listed.

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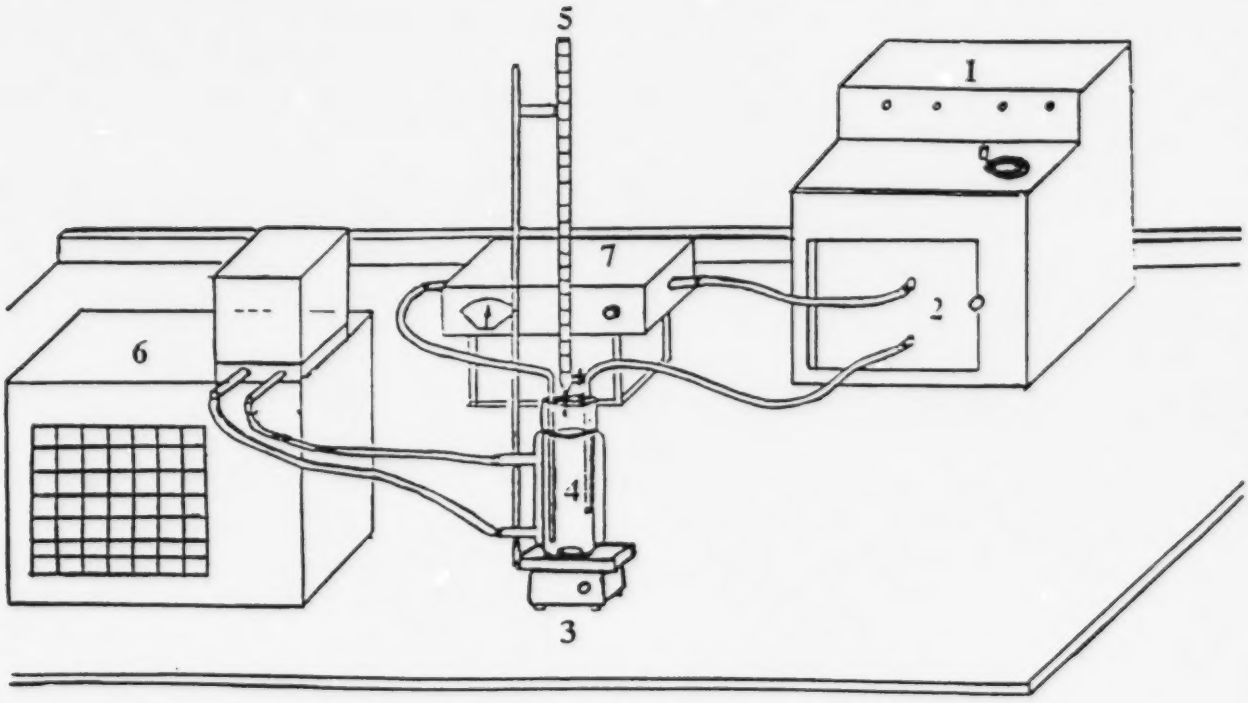
9.0 ACKNOWLEDGMENTS

The project participants (Cleary, Day, Gupta, Larkin, Lindsay, Murray, Thompson, Wiggin and Young) wish to acknowledge the advice and chemical samples received from Sydney Shapiro of Akzo Chemicals Inc.

CAHTFRG members who provided project planning support and guidance include Dr. Michel Boulanger of the Centre de Recherche du Quebec; Terry Graham of the Sunwell Engineering Company; Dr. Marc Paradis of Laval University; Dr. Keith Snelson of the National Research Council of Canada; and Dr. Chris Snoek of Atomic Energy of Canada Limited.

FIGURE 1: Flowthrough Nephelometer System for Detecting Micelle Formation (cmc) and Transformation (mtc)



1. Turner 111 Filter Fluorometer
2. Continuous Flow Door Attachment
3. Magnetic Stirrer
4. Sample Vessel
5. Burette
6. Constant Temperature Bath
7. Circulating Pump

FIGURE 2: Critical Micellar Concentrations (cmc)
and Micellar Transformation Concentrations (mtc)
for n-Alkyl TMASal Solutions in Water

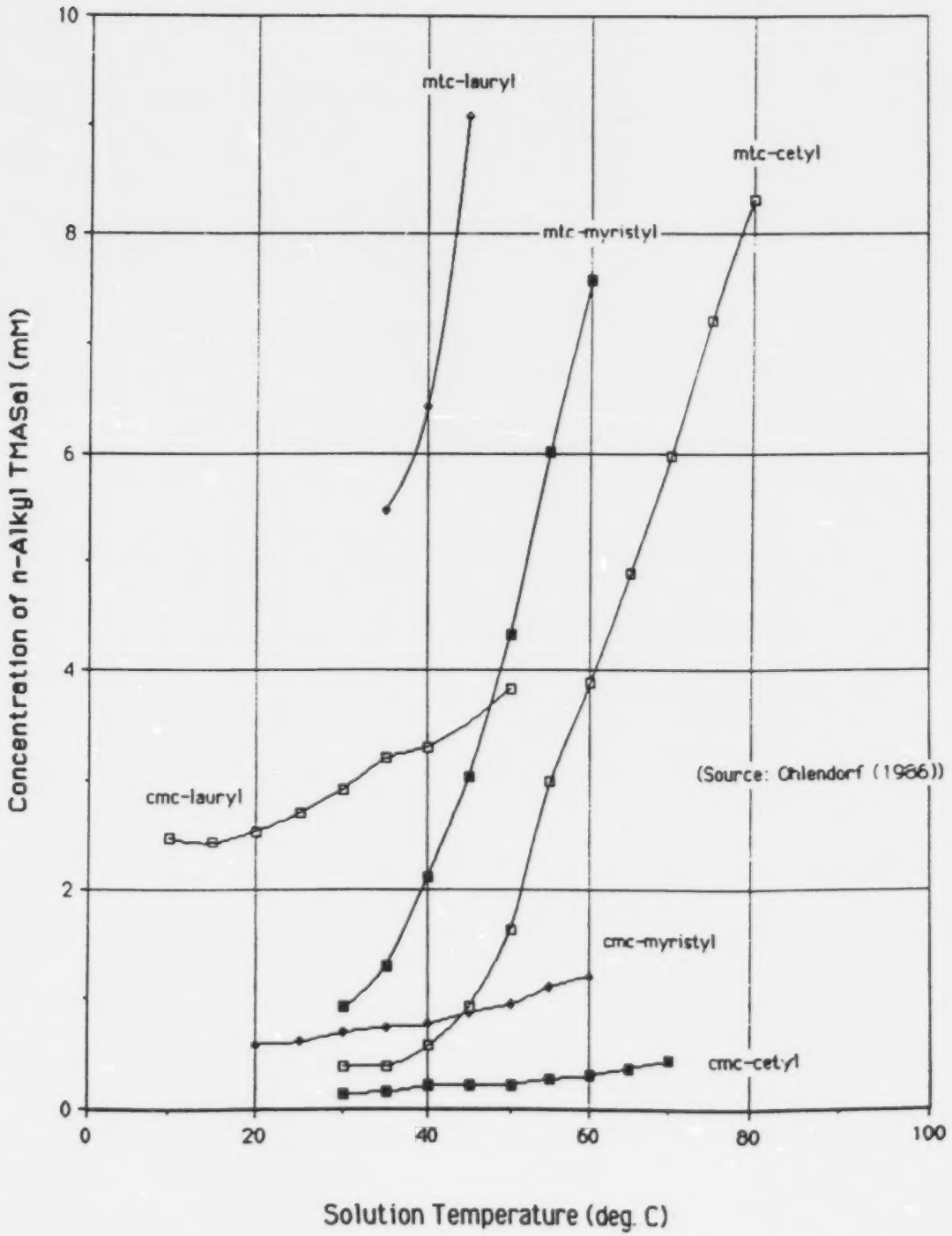


FIGURE 3: Rodlike-to-Spherical Micelle Transformation
(6 mM solutions of n-alkyl TMASal in water)

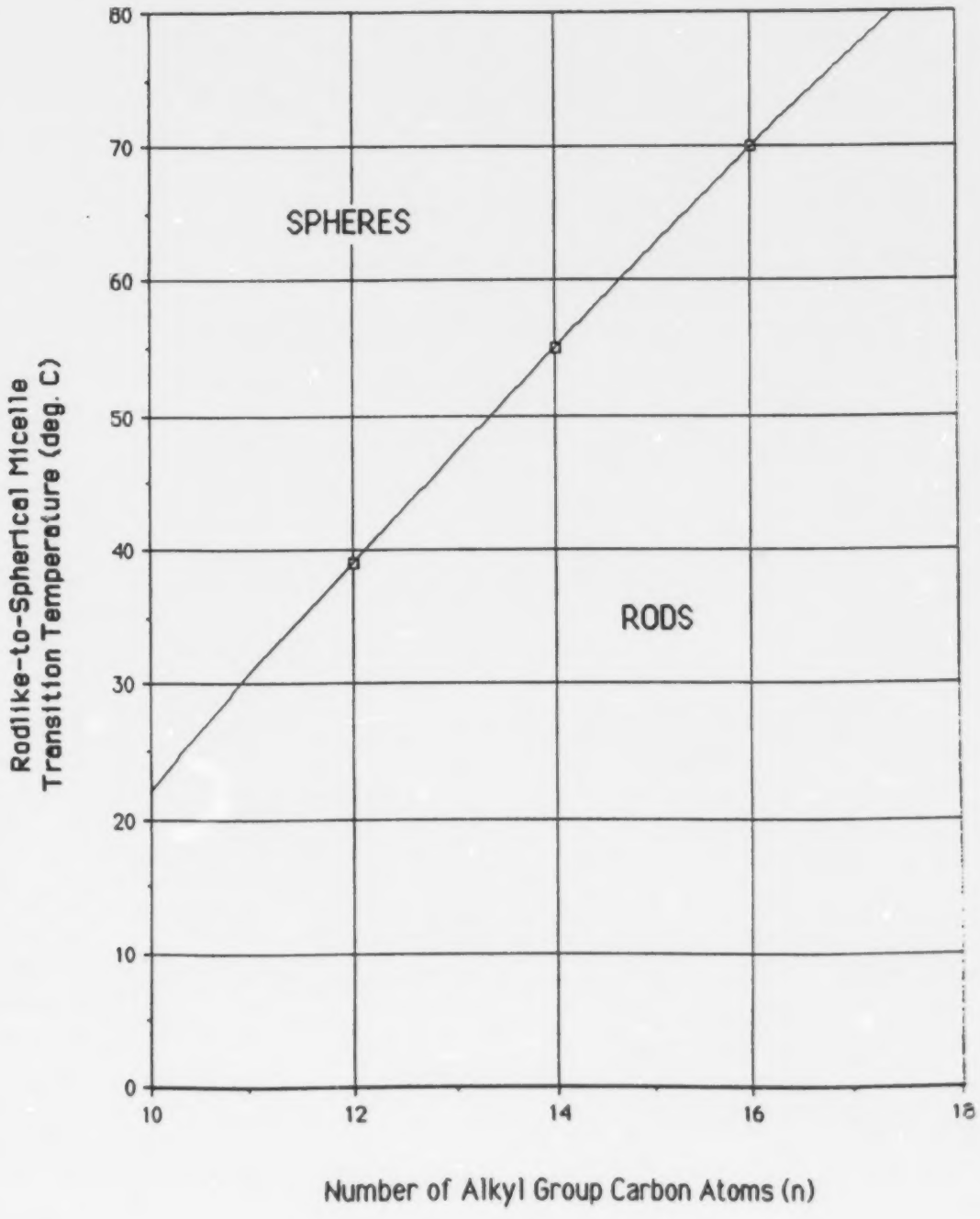
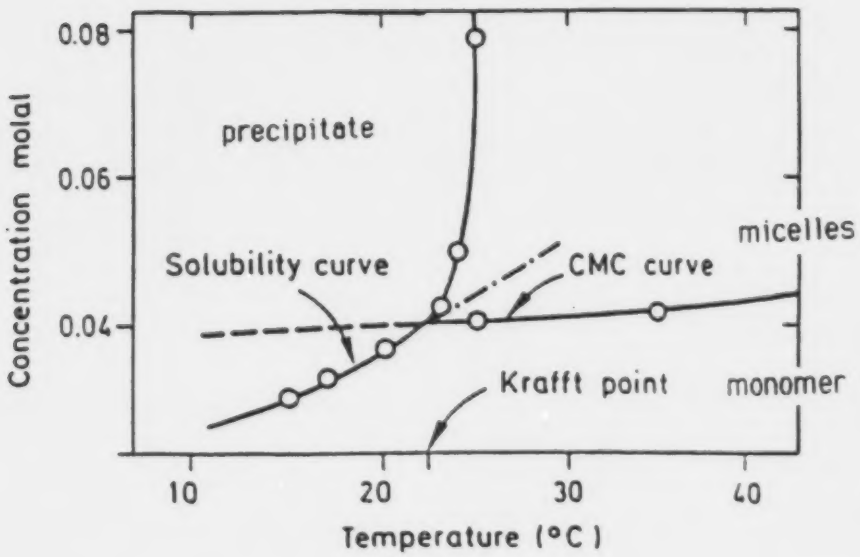


FIGURE 4: Micelle Formation and Transformation



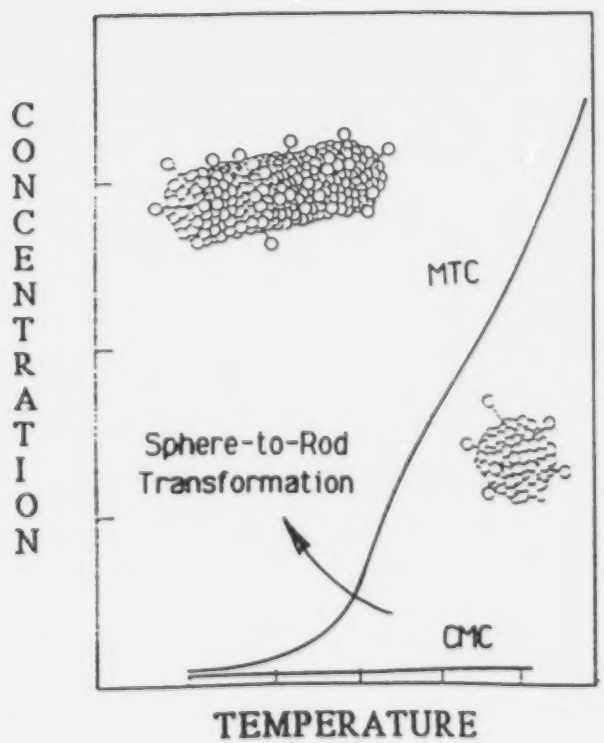


FIGURE 5: Dual-Tube Turbulent Flow Rheometer System

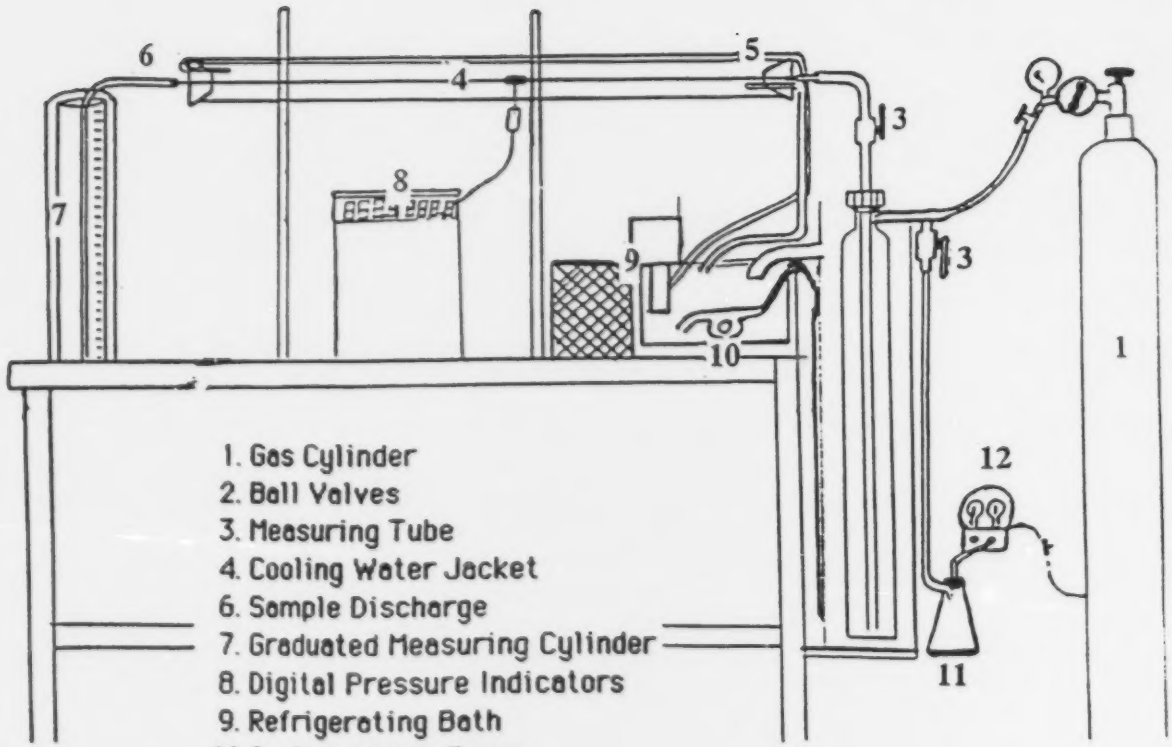


Figure 6: Flow Rate Enhancement by
n-DTMA 2,6-DHB and n-DTMA 4-MSal
(in 10% w/w e.g. solution at -2 deg. C)

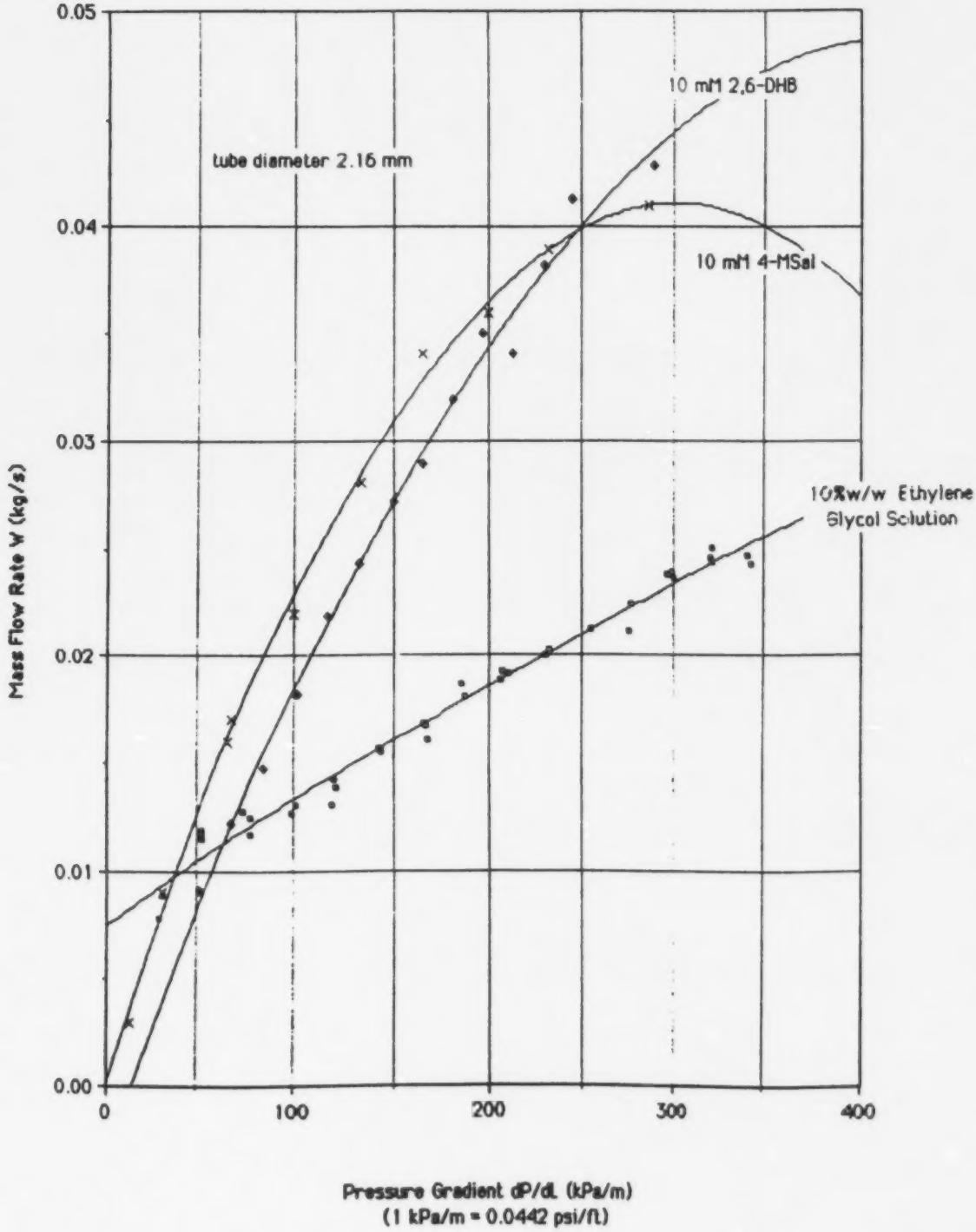


Figure 7: Effect of n-DTMA 2,6-DHB Dosage Level on Flow Rate Enhancement (in 10% w/w e.g. at -2 deg. C)

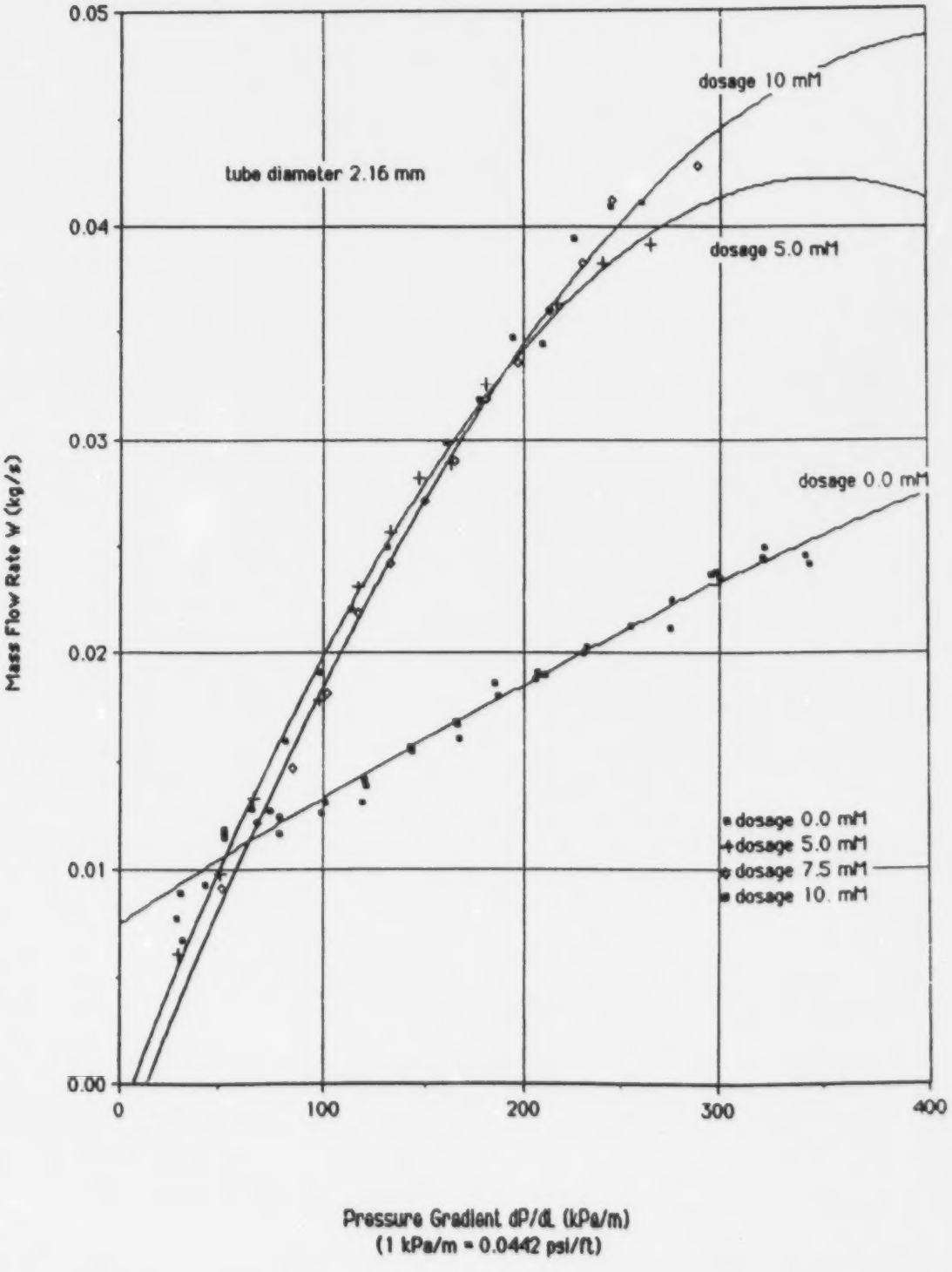


Figure 8: Percentage Flow Rate Increases
Produced by the n-DTMA 2,6-DHB Additive
(in 10% w/w e. g. solution at -2 deg. C)

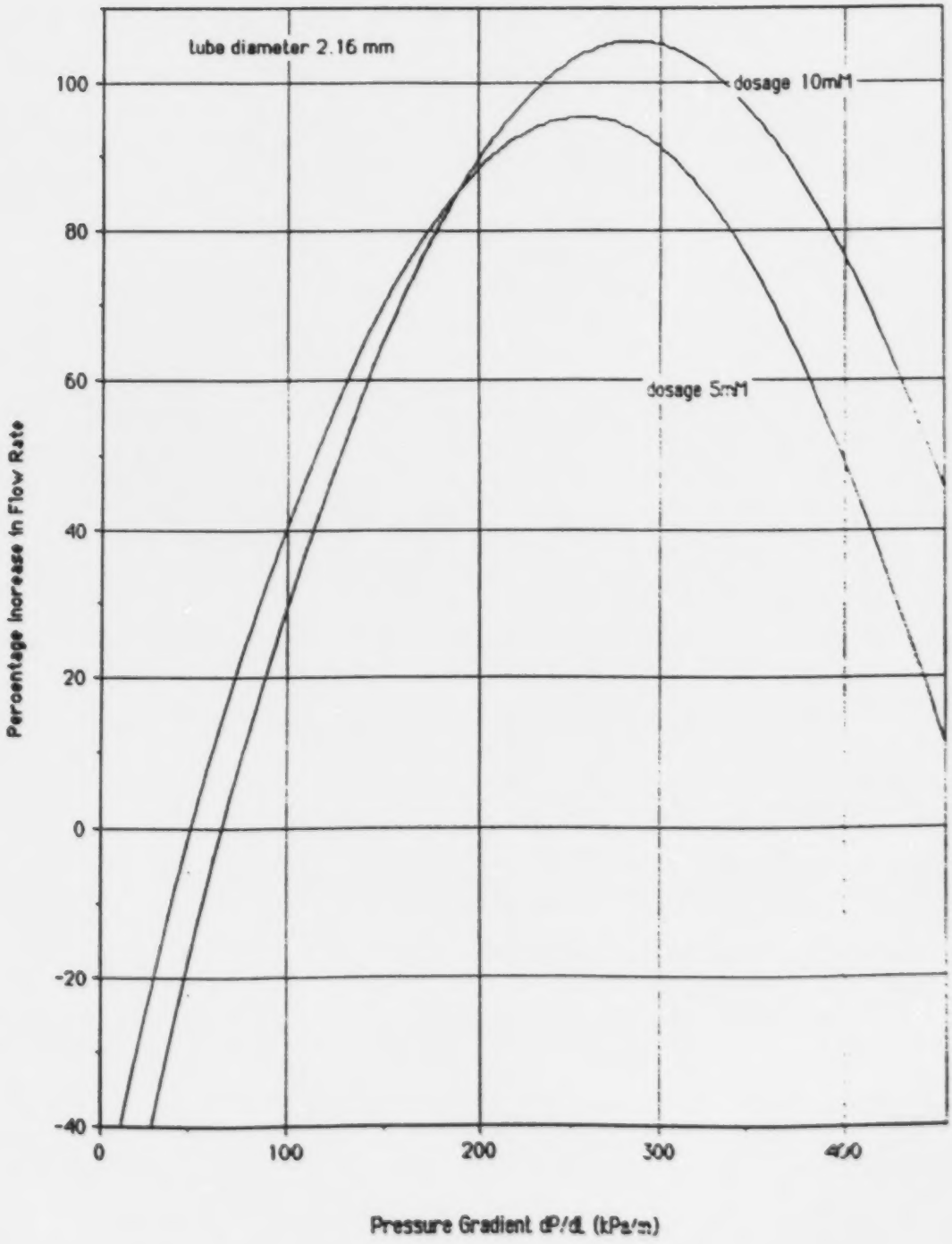


Figure 9 : Effect of n-DTMA 2,6DHB Dosage Level
on Darcy Weisbach Friction Factor

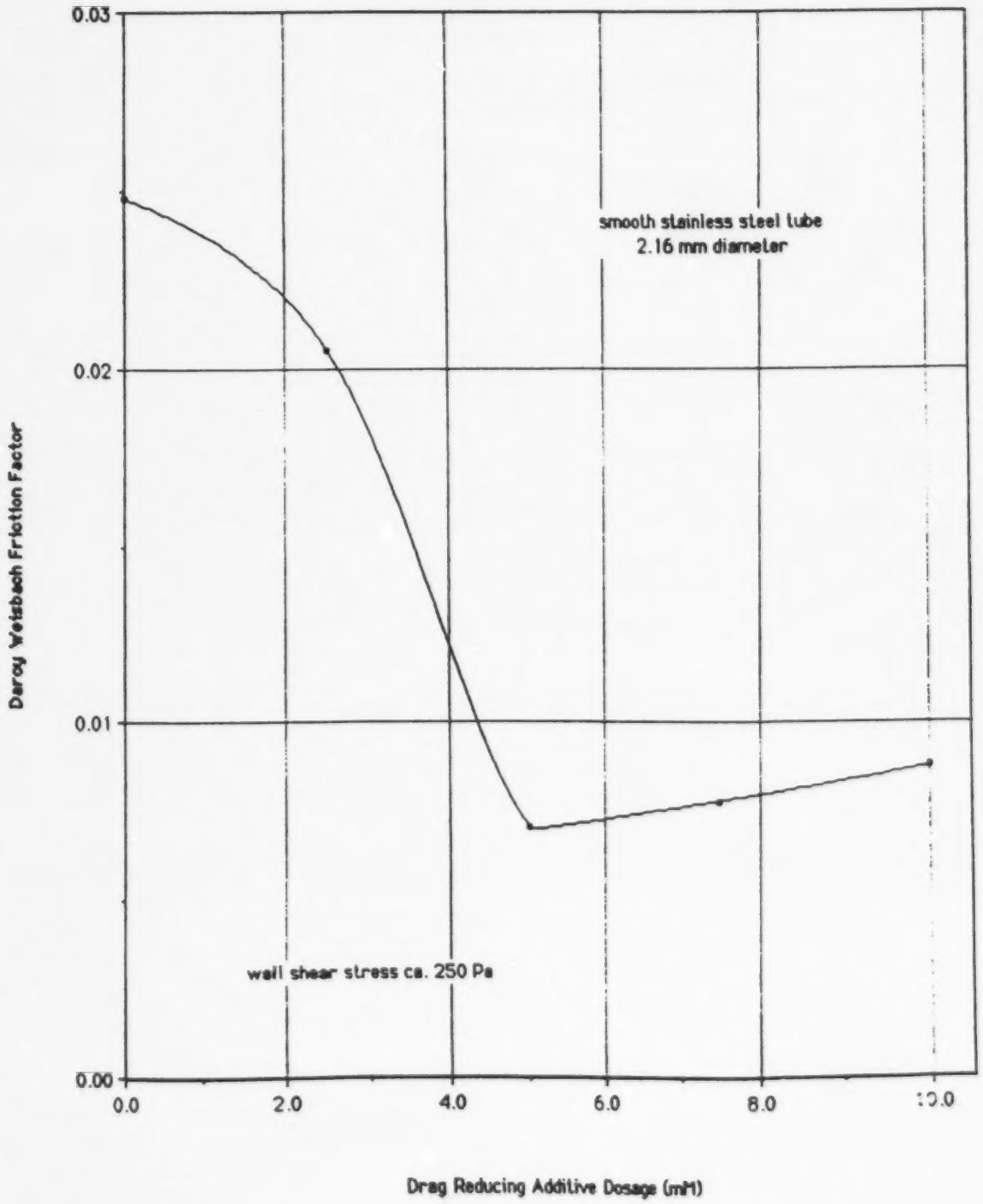


Figure 10: Friction Factor Reduction
by a 5 mM Dosage of n-DTMA 2,6 DHB
(in 10% w/w e. g. solution at -2 deg. C)

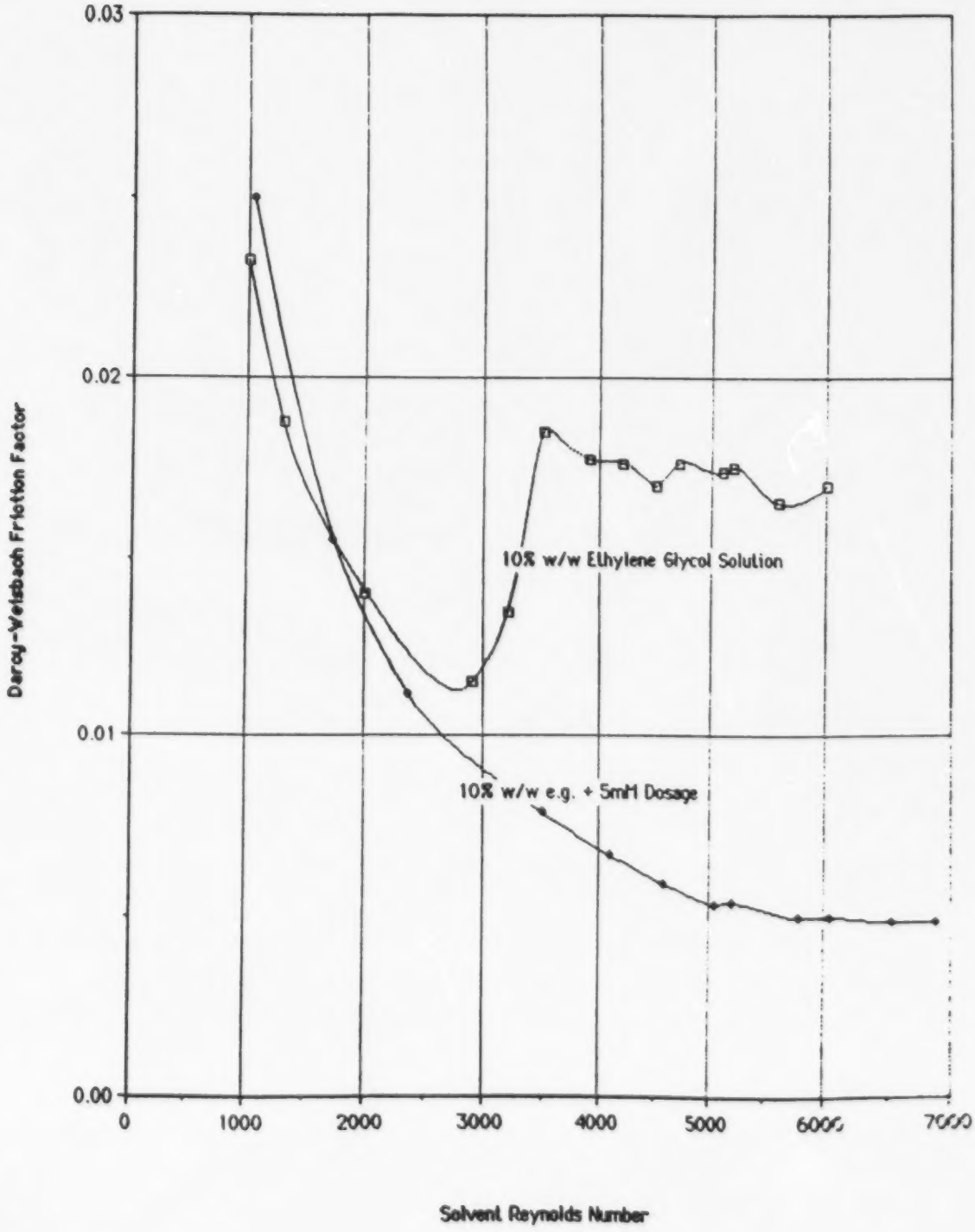


Figure 11: Effects of Temperature and Counterion Concentration on Flow Rate Enhancement by n-DTMA 2,6-DHB

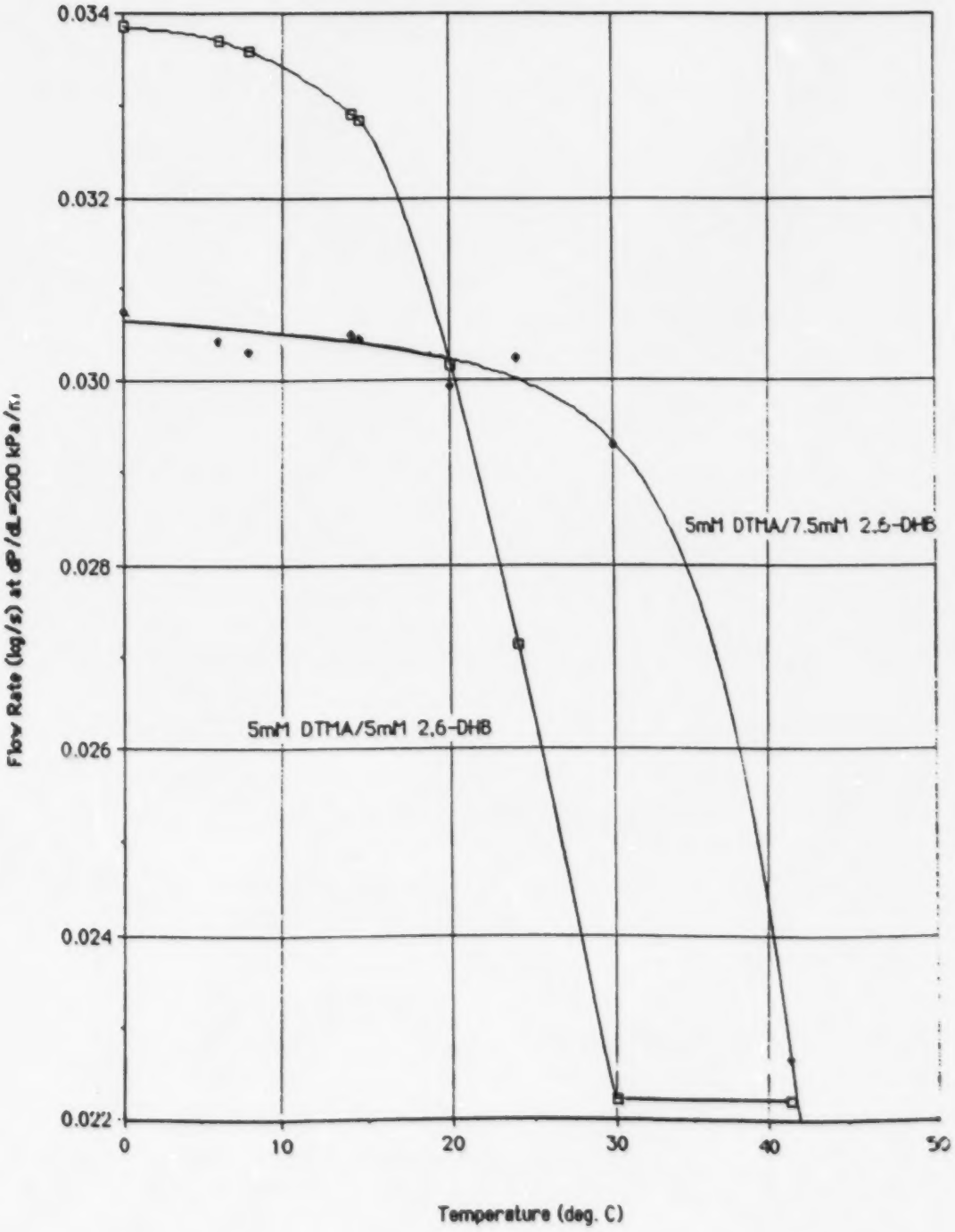
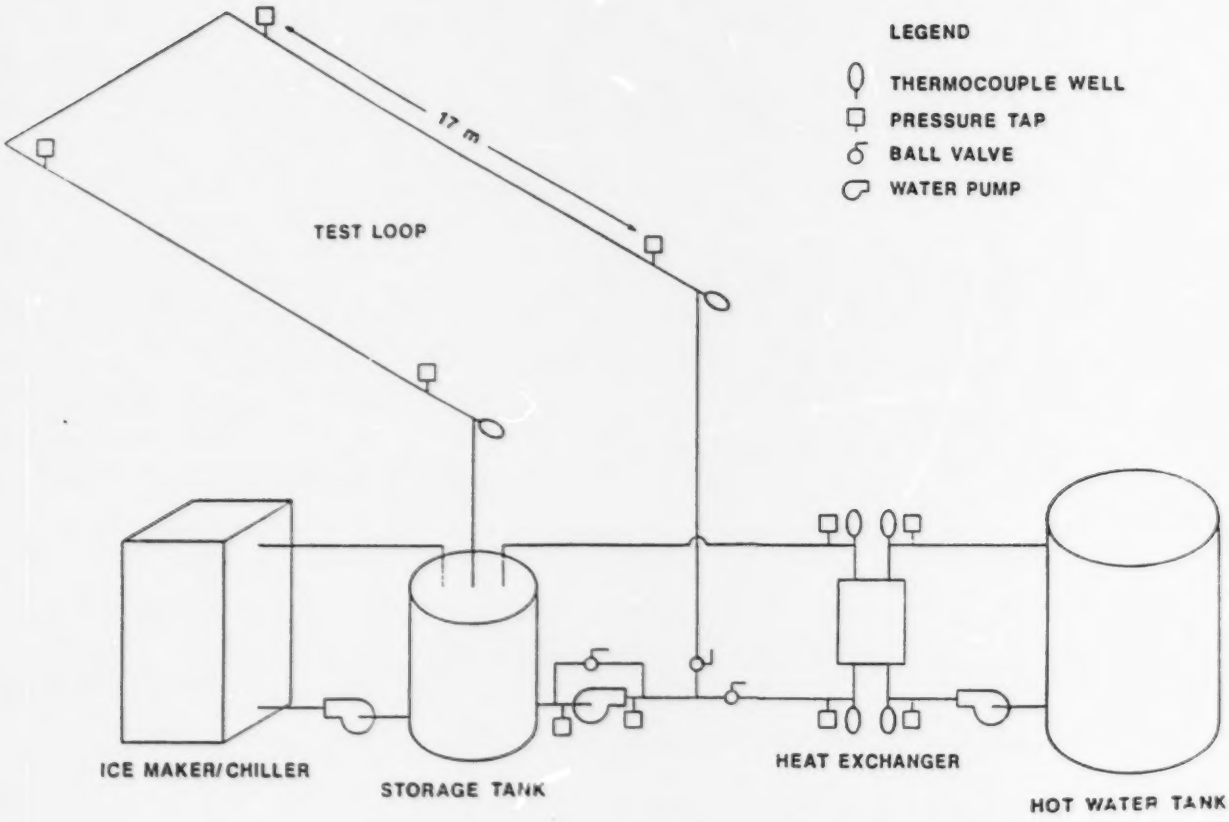
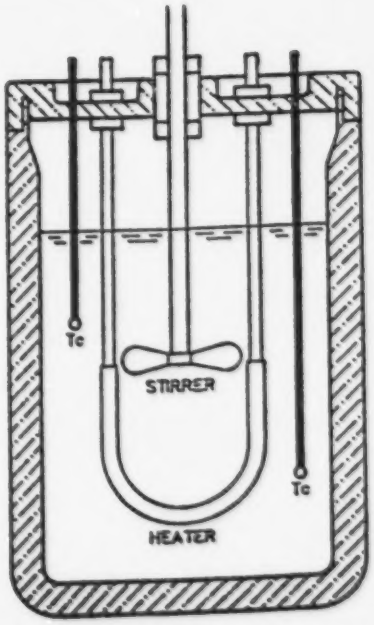


Figure 12: Ice Slurry Flow and Heat Transfer Test Facility



SCHEMATIC DIAGRAM OF ICE SLURRY TEST FACILITY



Tc – THERMOCOUPLE

Calorimeter for Measuring Slurry Ice Content

Figure 13: Flow Resistance Behavior - 25 mm Pipe Section - Freezing Run

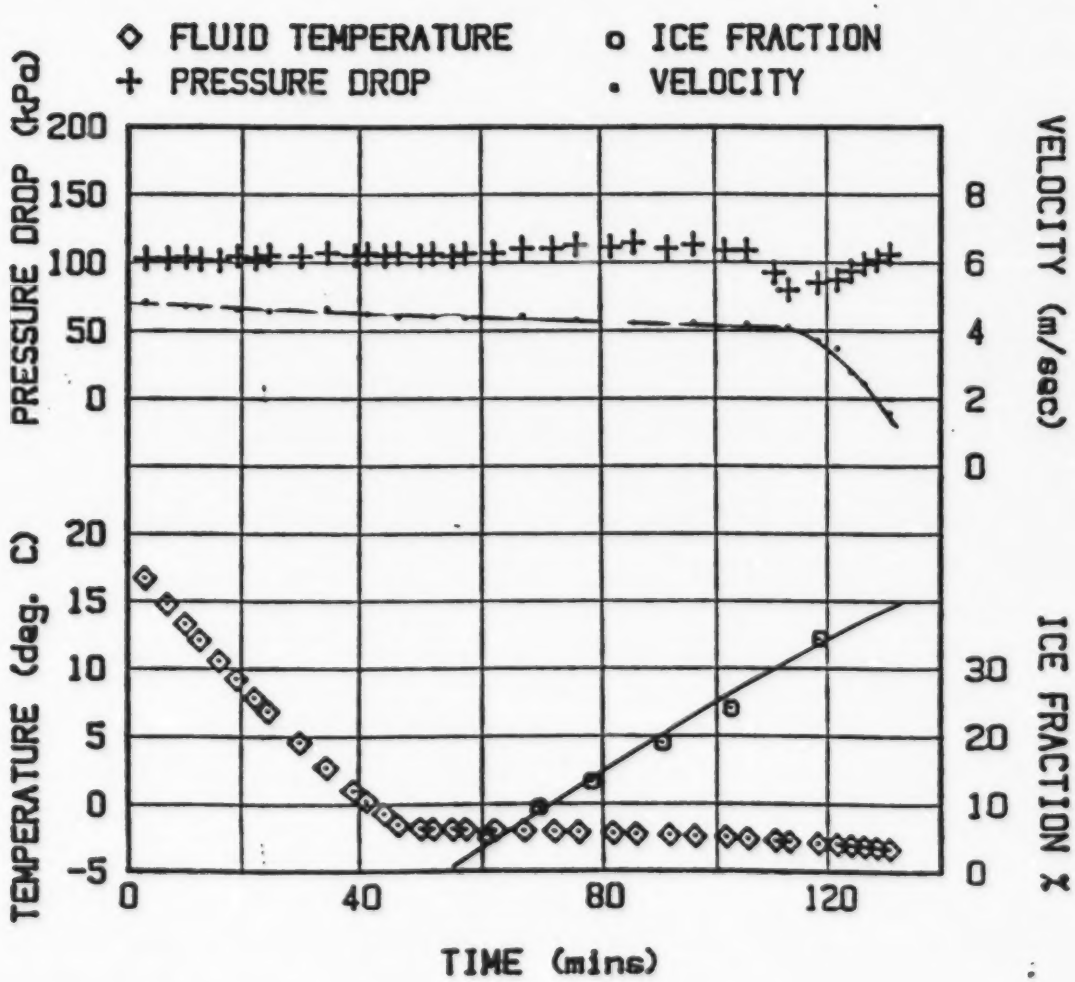


Figure 14: Flow Resistance Ratio - 50 mm Pipe Section - Freezing Run
(slurry and chilled water flow resistances corrected to the same viscocity and to a velocity of 3 m/s)

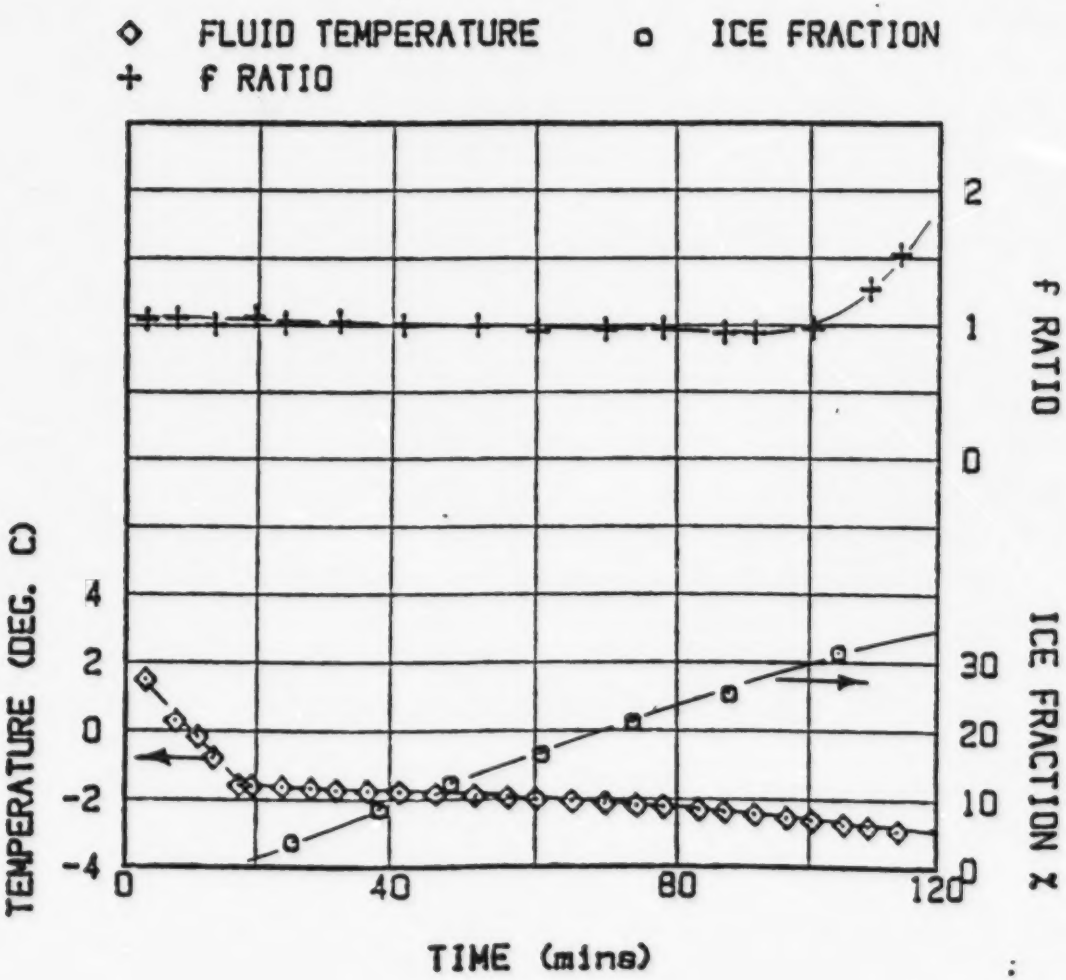


Figure 15: Critical Ice Concentration vs. Flow Velocity and Pipe Diameter

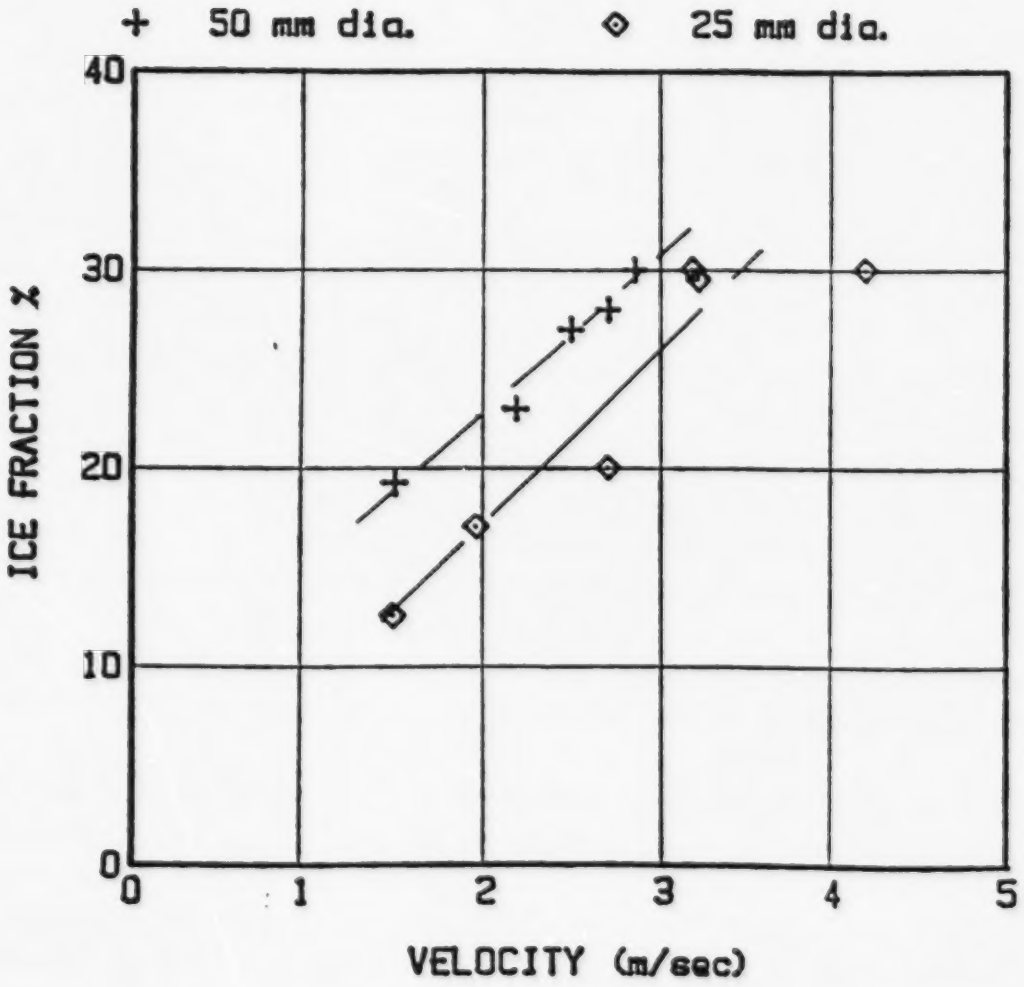


Figure 16: Heat Transfer Performance - Plate Heat Exchanger - Freezing Run

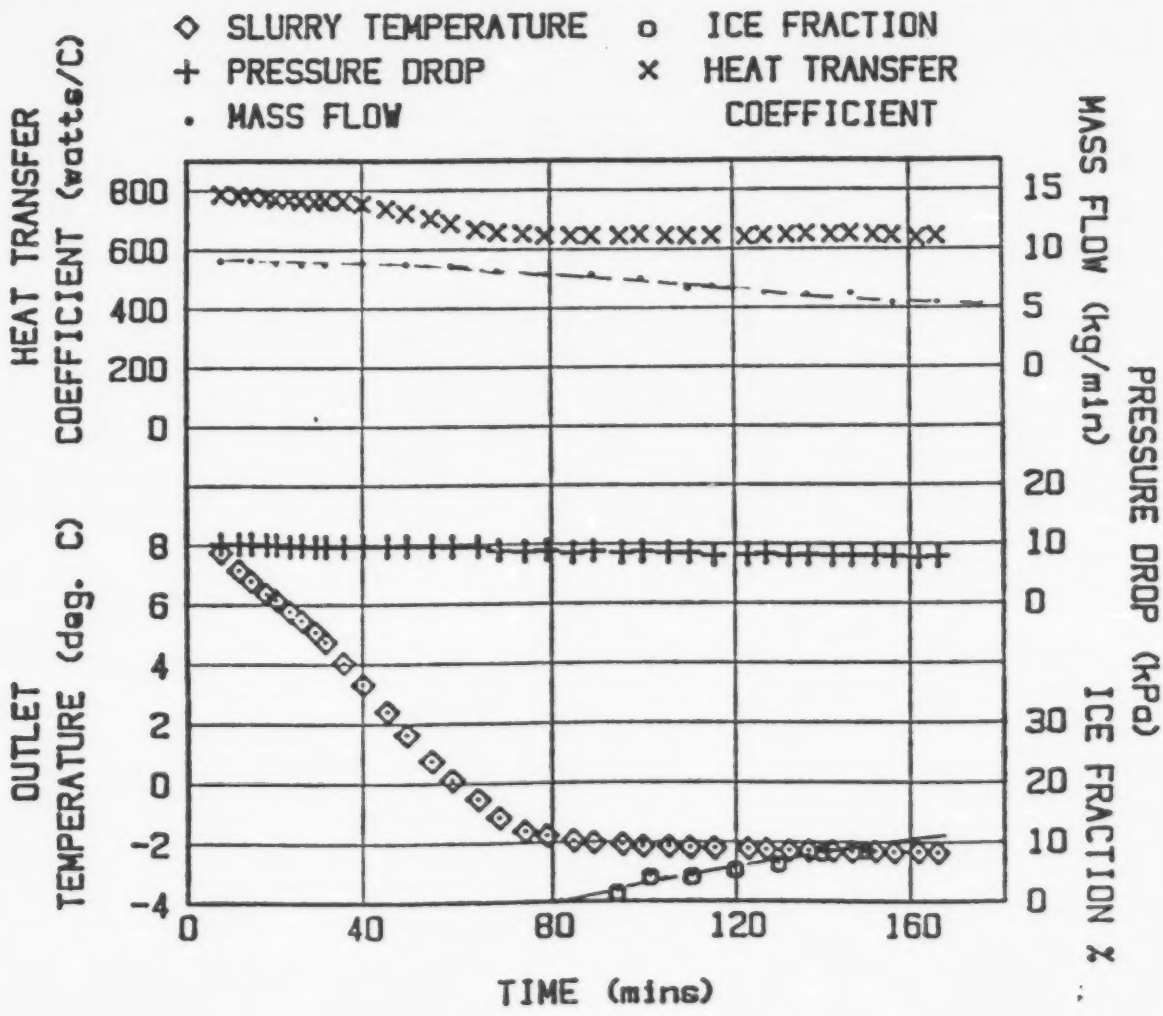


Figure 17: Heat Transfer Performance - Plate Heat Exchanger - Freezing Run
(pressure drop and heat transfer data corrected to a slurry flow rate of 9.1 kg/sec)

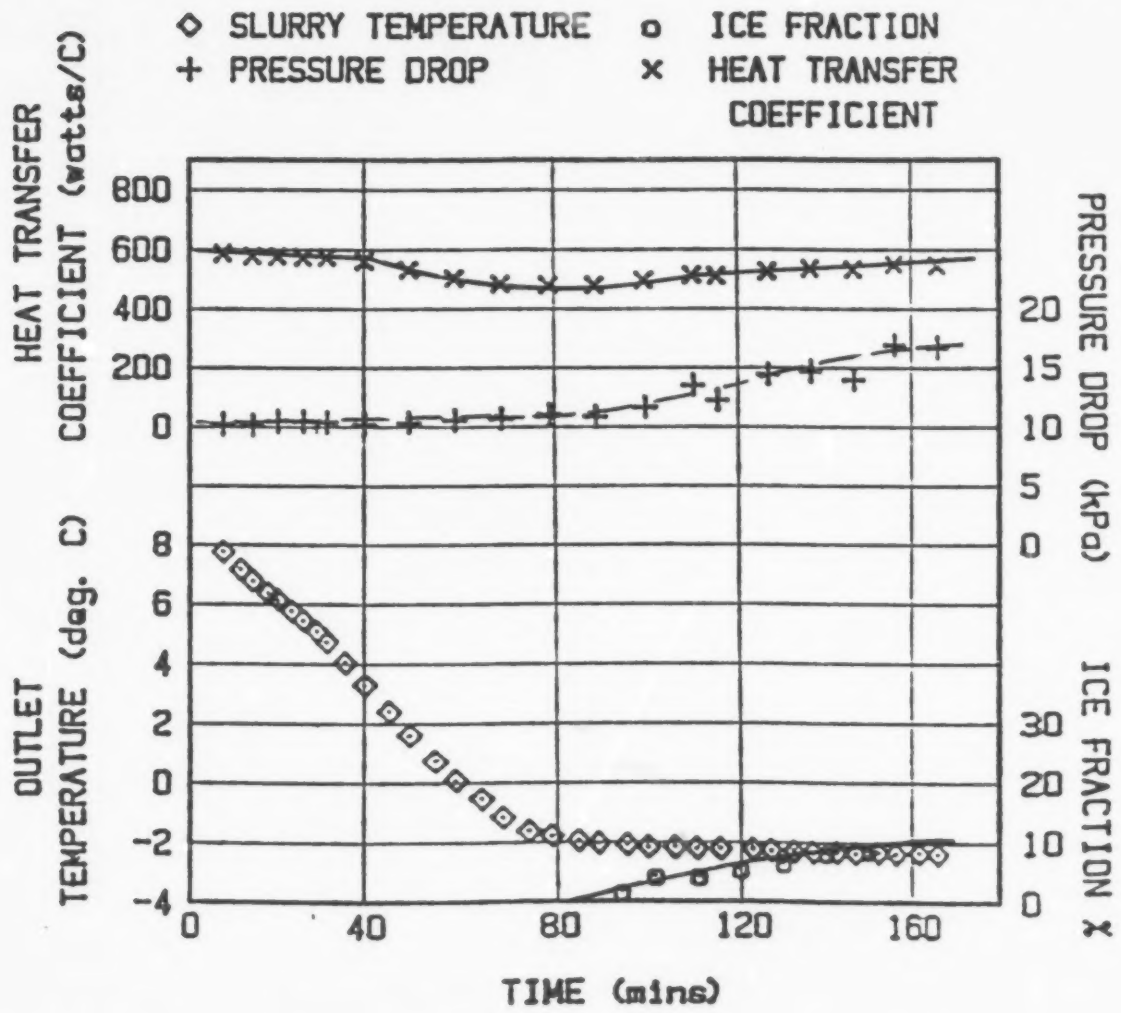


Figure 18: Heat Transfer Performance - Plate Heat Exchanger - Melting Run
(pressure drop and heat transfer data corrected to a flow of 36 kg/sec)

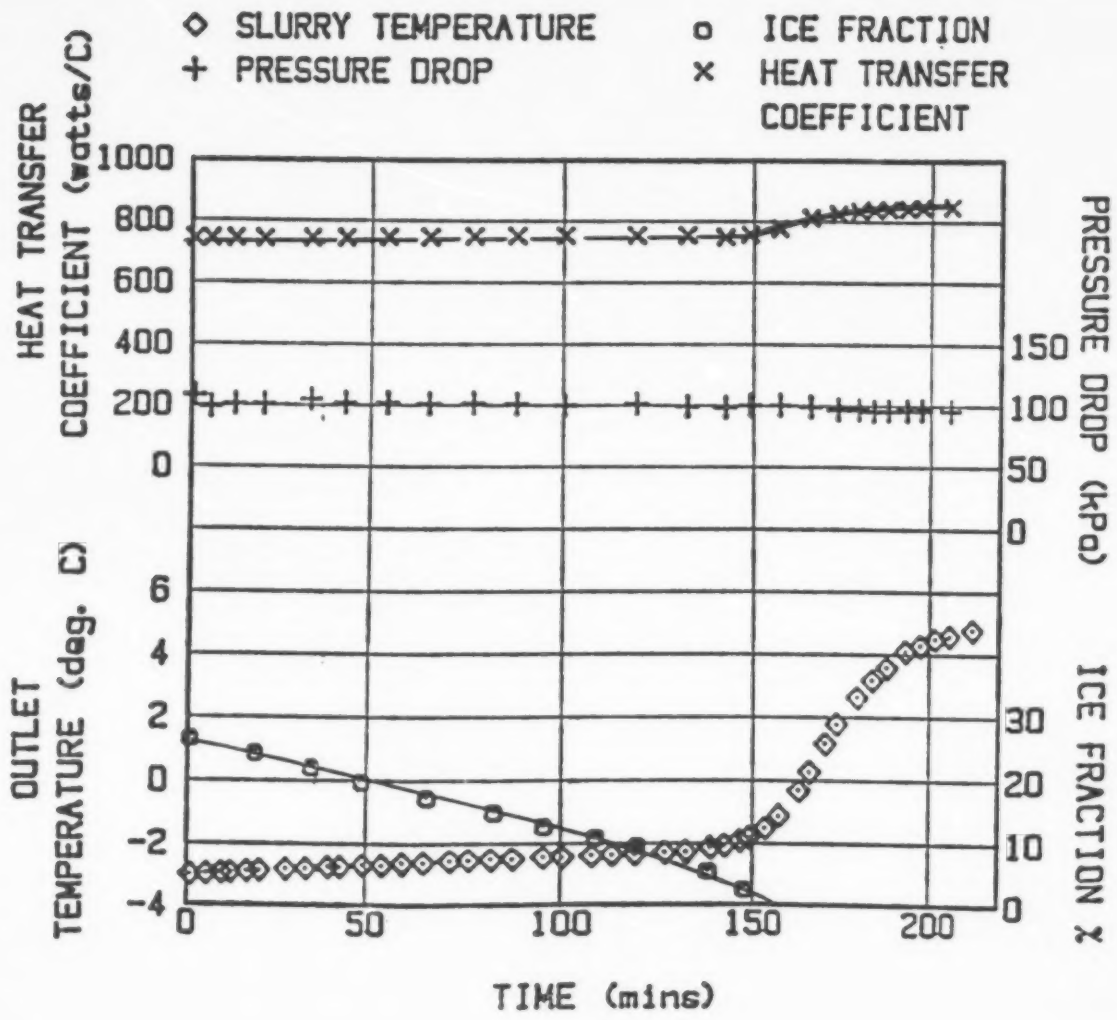


Figure 19: Heat Transfer Performance - Shell and Tube Heat Exchanger
(data corrected to a tube (slurry) side flow rate of 12.3 kg/sec)

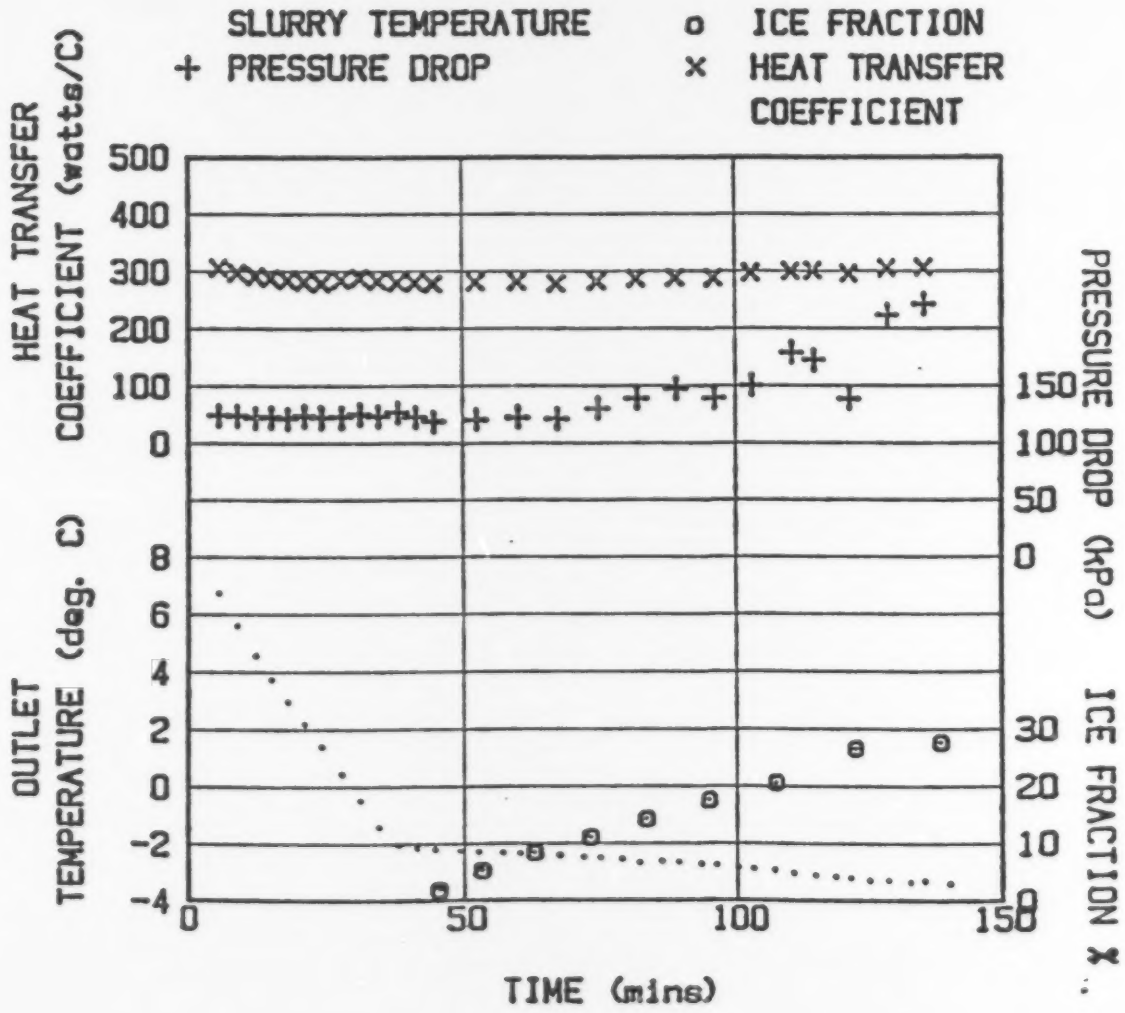


Figure 20: Ice Particle Size Distribution and Growth with Aging

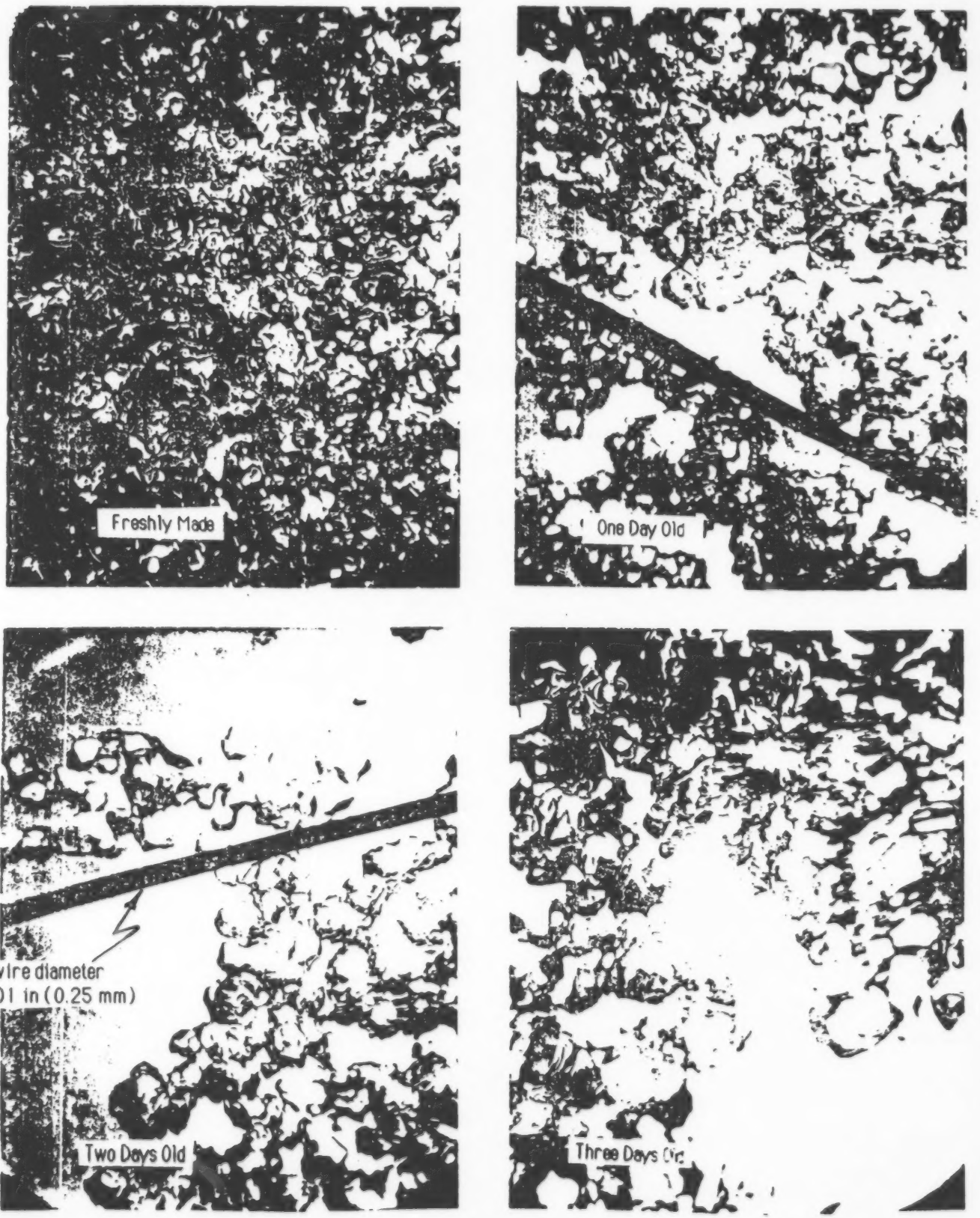


Figure 21: Effect of Interfacial Area on Heat Transfer Rate

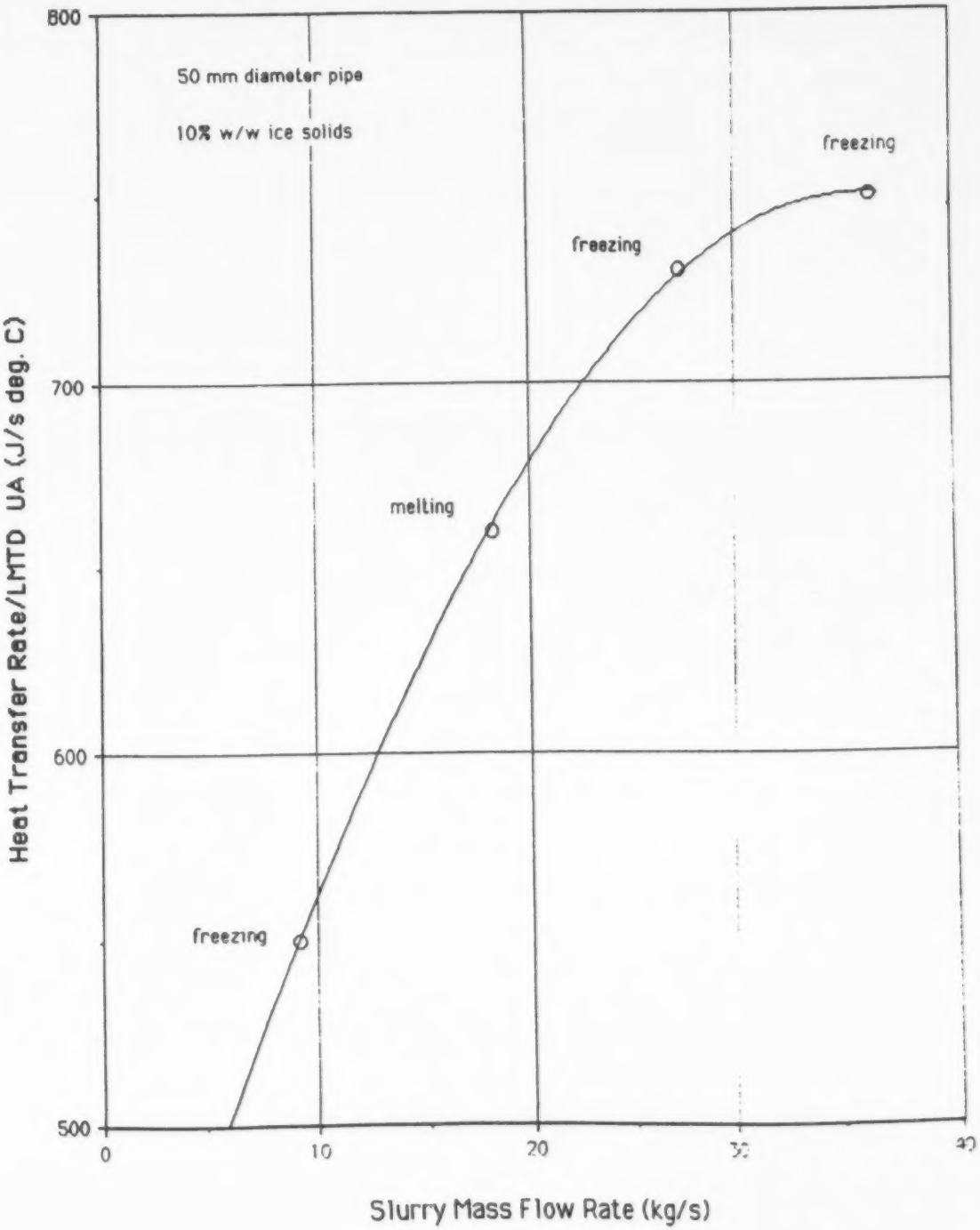


Figure 22: Comparison of Methods of Measuring Ice Solids Concentration

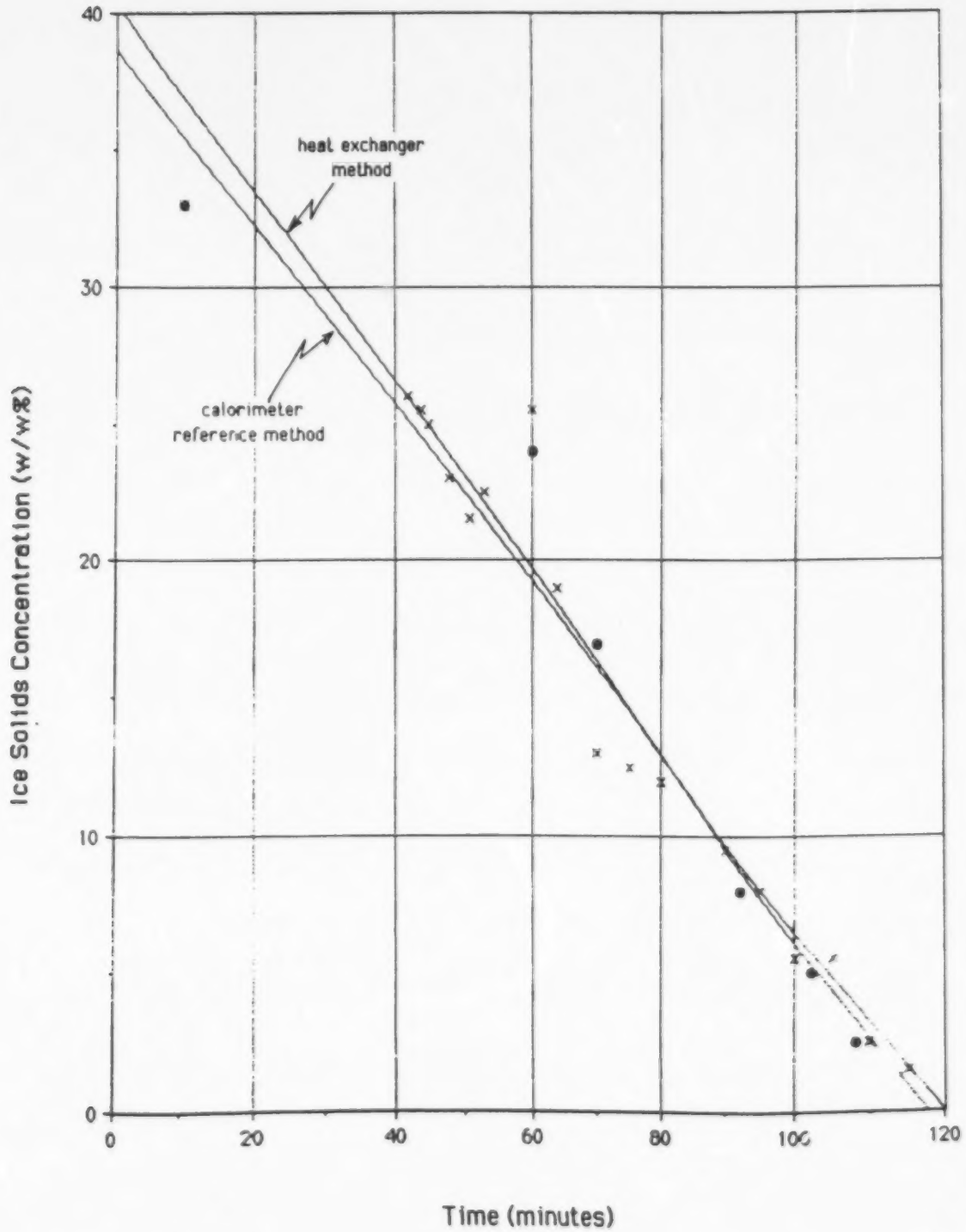


Figure 23: Effects of Drag Reducing Agent Addition
on Pipeline and Heat Exchanger Pressure Gradients